











# **TEXTILE CHEMISTRY**



# **TEXTILE CHEMISTRY**

**AN INTRODUCTION TO THE CHEMISTRY  
OF THE COTTON INDUSTRY**

**BY**

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**WITH 240 DIAGRAMS**

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## PREFACE

**A**Dvanced manuals on various branches of the textile industry are fairly numerous, but often the proper appreciation of them is considerably impaired by a lack of elementary chemical knowledge on the part of students and workers who are most interested in their contents.

This book is intended to supply this deficiency.

For several years the matter incorporated in this volume has been the basis of instruction introductory to the systematic study of the technical processes of Sizing, Bleaching, Dyeing and Finishing, and the Chemistry of Mill Stores and Materials. It has been put into permanent form only after considerable experiment and experience.

The Syllabus of the Union of Lancashire and Cheshire Institutes in Textile Chemistry is admittedly framed on it and the ground covered includes the subjects of the Syllabus of the City and Guilds of London Institute in "Chemistry as Applied to the Cotton Industry," Subject 28E in the programme of the Department of Technology; it is hoped, therefore, that the book will be particularly suitable for classes in Technical Schools conducted under the regulations of these bodies.

But it should appeal also to the young studious cotton operative who desires to increase his technical knowledge, and to those in positions of authority in mills who wish to know something of the nature, preparation, and properties of the materials used in the various processes through which cotton must pass in its journey from fibre to marketable cloth.

The attempt has been made—it is hoped successfully—to produce a manual which shall serve not only as a textbook for a school course which includes lectures and practical work, but also as a complete laboratory manual for those who cannot obtain special practical instruction, and finally as a trustworthy guide for the private student.

The user hardly needs to be reminded that it should be worked through in an experimental manner; every exercise should be performed by, or demonstrated to, the student, and no attempt should be made to "cram up" the information it contains.

Several friends have been good enough to read and criticize the manuscript when ready for the press, of whom I wish to thank

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publicly Mr. Harold Hunter, of the Battersea Polytechnic; Mr. H. G. Leigh, of this College; and Mr. R. B. Duerden, of Nelson.

Many pieces of apparatus described in this book are new and original and not included in the usual trade catalogues of makers of Scientific Apparatus, but arrangements have been made with

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to supply the same, and all communications relating thereto should be addressed to them and *not* to the publishers or author.

Small cotton hanks and dyes for experimental purposes may be obtained from

**MESSRS. "COMMERCIAL LABORATORIES,"  
HART CHAMBERS, VICTORIA STREET,  
BLACKBURN. \***

Finally, I desire to express my gratitude to Dr. R. H. Pickard, F.R.S., for the opportunities he gave me, when he was Principal M. A. Hert, of developing this scheme of work in the day classes, and for the interest he took in it to make it successful.

F. J. COOPER

BLACKBURN, LANCS.

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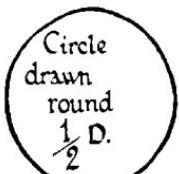
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## SECTION I

### I. HOW TO DRAW DIAGRAMS

THE correct representation of chemical apparatus is a very important preliminary to the study of chemistry. Very few lessons are complete unless accompanied by sketches or diagrams of the articles used in the various preparations.

In order to reduce to a minimum the time spent in drawing dia-



THE UNIT  
Fig 1

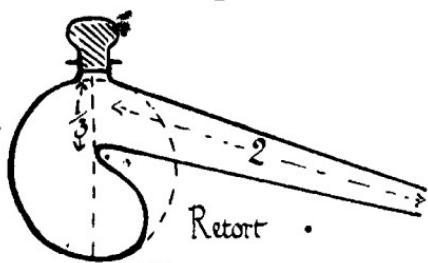


Fig 3



Fig 2

grams, and to ensure that they shall be fairly uniform, some *system* should be adopted. The following method will be found to be as satisfactory as any, and more so than most.

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All lines should be drawn first in pencil (where necessary with the aid of a ruler), and then the completed diagram inked in freehand throughout. Every diagram in this book has been produced in this manner.

Circles should be drawn by tracing round a halfpenny (Fig. 1), and the diameter of this circle should be considered as a unit of length upon which is based the dimensions of the figures.

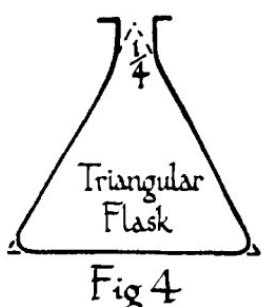


Fig 4

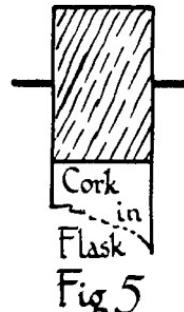


Fig 5



Fig 6

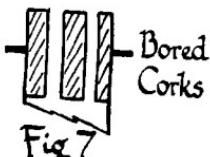


Fig 7

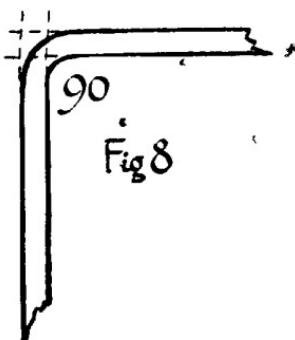


Fig 8

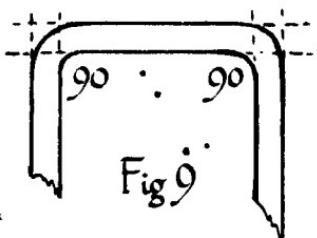


Fig 9

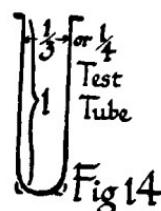
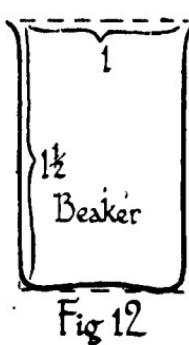
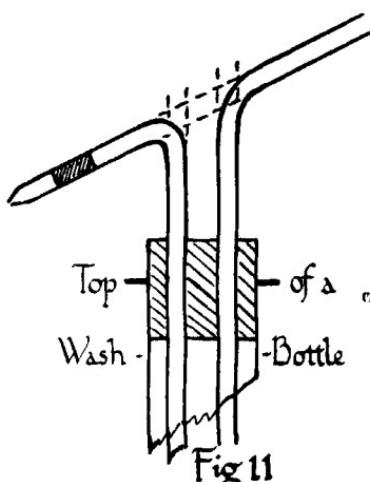
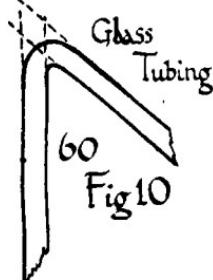
The flask is drawn, as shown in Fig. 2, by making the circle, drawing a vertical diameter, and continuing it one unit. Parallel lines are drawn on each side of this, the width between being equal to  $\frac{1}{4}$  of the unit length. Shoulders are put on where they meet the circle, the

bottom is formed by cutting a segment at the base, and the flange is formed by drawing short straight lines at right-angles.

The method for drawing a *retort* is shown in Fig. 3.

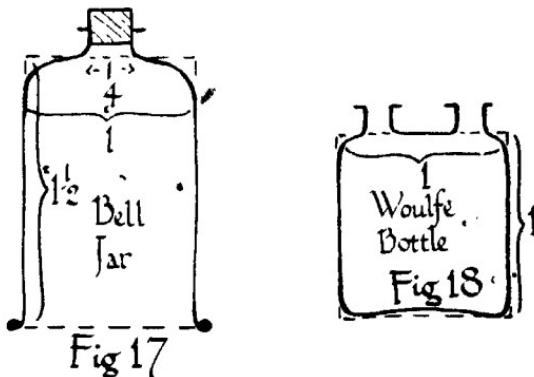
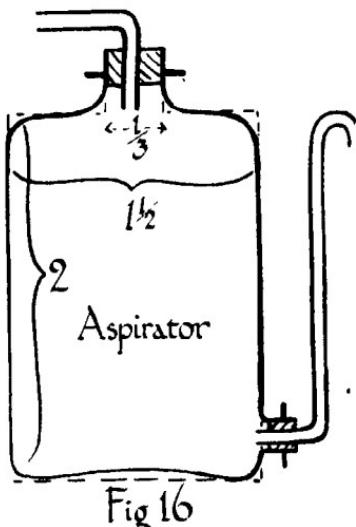
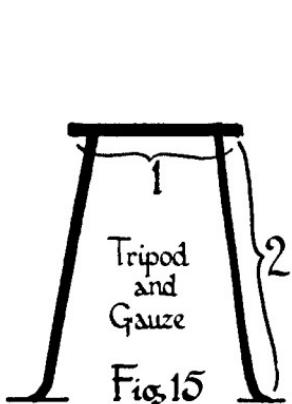
The *triangular flask* is evolved from an equilateral triangle of sides  $1\frac{1}{2}$  units long (Fig. 4).

*Corks* should be drawn with lines which are continuations of the neck of the flask, etc. (Fig. 5). Figs. 6 and 7 show (much enlarged) how *holes* should be represented in diagrams of corks.



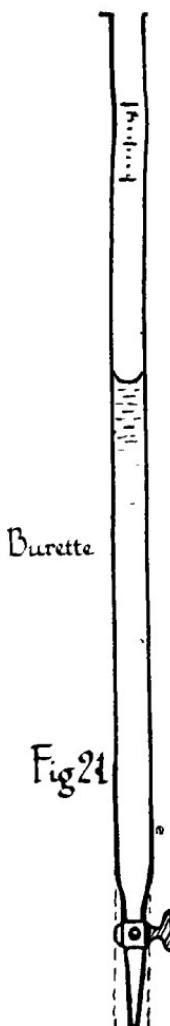
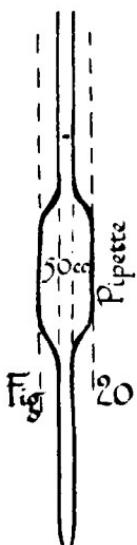
## TEXTILE CHEMISTRY

*Glass tubing and glass bends* should be drawn as two parallel lines, the corners being rounded off, the outside one last, as shown in Figs. 8, 9, 10. Fig. 11 gives the construction lines necessary for drawing the tubes in a laboratory *wash bottle*.



The *beaker* (Fig. 12), *gas jar* (Fig. 13), *test tube* (Fig. 14), and *tripod* (Fig. 15) are all very easily drawn.

Rectangles form the skeletons for the *aspirator* (Fig. 16), *bell jar* (Fig. 17), and *Woulfe bottle* (Fig. 18).



Parallel lines form the basis upon which are constructed the following: gas jar collecting a gas by displacement, pipette, burette, Liebig condenser, and retort stand (Figs. 19 to 23).

Fig. 24 illustrates the correct way to represent the *bunsen birner*. The arrangement when heating a crucible supported by a *pipeclay*

## TEXTILE CHEMISTRY

*triangle resting on a tripod* is given in Fig. 25. The combination shown in Fig. 26 represents an *evaporating dish* placed on a *sand bath* being heated by a bunsen burner provided with a rose.

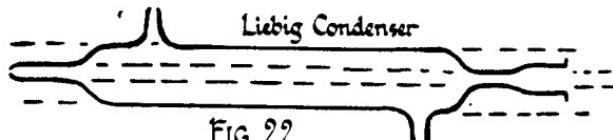


FIG. 22

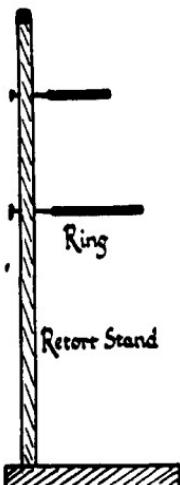


FIG. 23

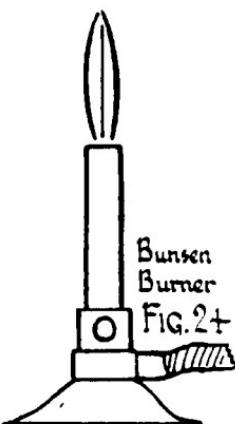
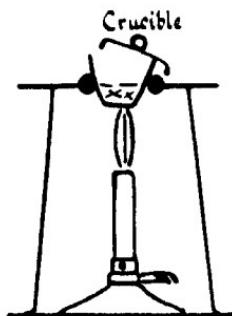
Bunsen  
Burner  
FIG. 24

FIG. 25

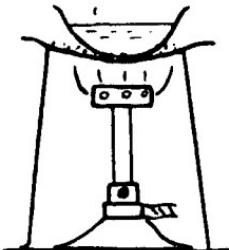


FIG. 26

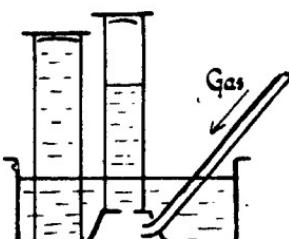


FIG. 27

If a gas is being collected by displacement of water in a *pneumatic trough*, the diagram is drawn as shown in Fig. 27. A gas

## CHEMICAL DIAGRAMS

jar is standing on a *beehive shelf*. Liquid is always represented as a continuous straight line for the surface, with dotted lines under it.

Other diagrams frequently required are:—*Wurtz or distillation*

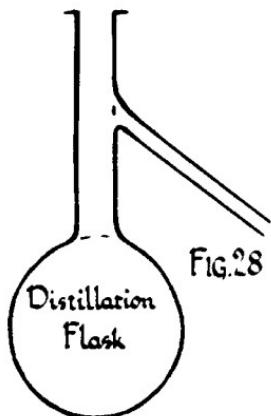


FIG. 28



FIG. 29

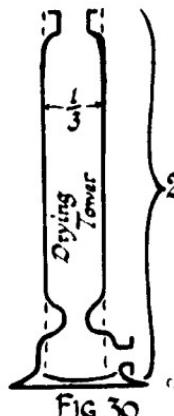


FIG. 30

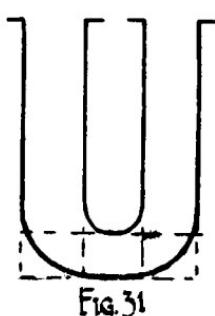


FIG. 31

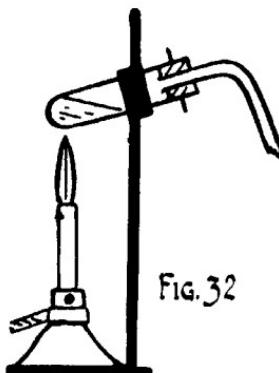
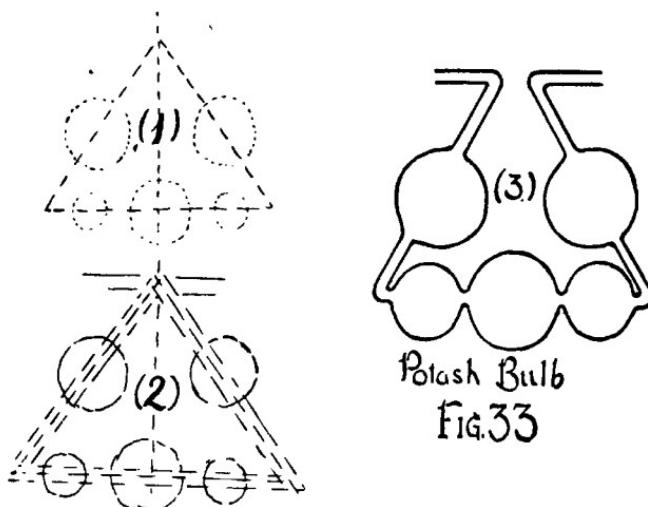


FIG. 32

*flask* (Fig. 28), *acid or thistle funnel* (Fig. 29), *drying tower* (Fig. 30), *U tube* (Fig. 31), *retort stand and clamp supporting boiling-tubes*, etc. (Fig. 32).

The method of construction of a *potash bulb* is shown in three stages in Fig. 33 (1, 2, 3).



## II. CHEMICAL TOOLS

Every trade has its tools. In chemistry we call them apparatus. Those described in the following pages are of very general use and necessary for the work which follows.

**1. The Balance.** This is the most important piece of apparatus a chemist possesses; without it he can do nothing: in fact chemistry was not a science till its workers used a balance.

Good and accurate balances are now easily obtainable. A modern form of student's balance is shown in Fig. 34.

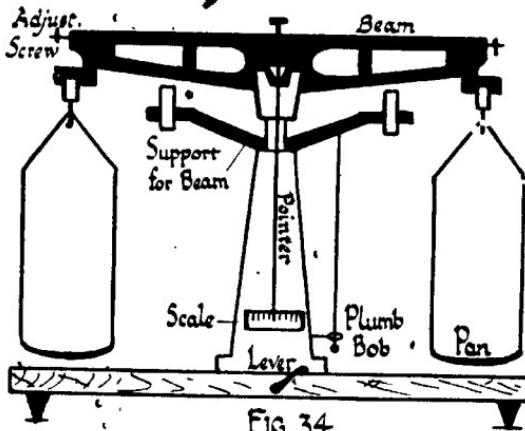


FIG. 34

A box of weights containing grams and fractions of a gram must always be provided for use with a balance. Figs. 35 and 38 show in section the usual shape of brass gram weights, which are arranged in a box as shown in plan in Fig. 36. Note that the 20's and 2's are duplicated. A groove is cut in front for the tweezers (Fig. 37) with which weights are *always* moved.

Fractions of a gram are often kept in a separate box or a separate compartment. They are usually numbered in milligrams and range from 500 to 10.



FIG. 35

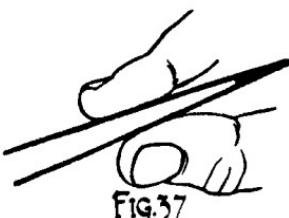


FIG. 37

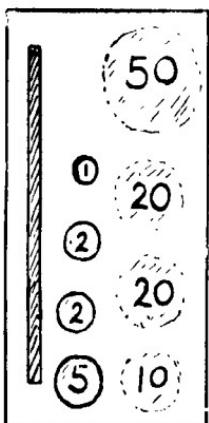


FIG. 36



FIG. 38

They are made of platinum, German silver, or aluminium in the form of foil (Fig. 39) or wire bent into various shapes. Fig. 40 is a new and very good form in which the wire is so bent that the weight in milligrams is seen at a glance.

If it is required to weigh more accurately than to 10 mg. a *rider* is used on the beam. This article (Fig. 41) is a stirrup of aluminium wire which weighs exactly 10 mg., and if placed in the pan of the balance acts as a weight of 10 mg. But if it be placed *on* the beam it only weighs that amount if placed at the end.

If the beam be divided into 10 equal spaces between the knife-

edge and the pan, as in Fig. 42, and the rider is placed on one of these divisions, it will weigh less than 10 mg. If on division 1, it will weigh 1 mg.; on division 2, 2 mg.; on division 3, 3 mg., etc.

#### THE PROCESS OF WEIGHING WITH A BALANCE

1. Test the balance to see if it is accurate—that is so when the pointer swings an equal distance on each side of the zero mark.
2. Substances must always be placed on a watch glass or other receptacle—*never on the bare pan*.
3. Weights should be placed on the right-hand pan. Commence with the largest; remove it if it is too heavy, allow it to remain if too light; and add in descending order, *missing none*.

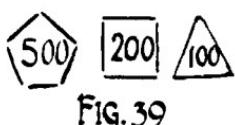


FIG. 39

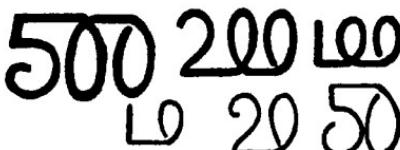


Fig 40



Fig 41

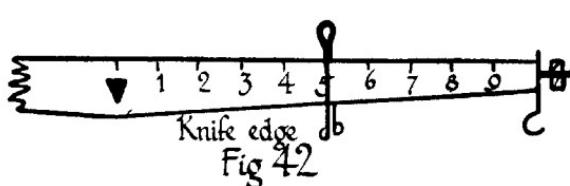


Fig 42

4. Do not put anything on or remove anything from the pans, whilst the balance is swinging—always bring it to rest first.

5. When a correct balance is obtained count up, first the number of whole grams, then the number of milligrams, and write down with a decimal point between, e.g. 25 grams 830 mg. = 25.830 grams.

If the mg. had amounted to 83 only, a cipher would have been entered in the hundred column, e.g. 25.083.

#### EXERCISES

1. Find the weight of a crucible and lid.
2. Weigh a beaker or an evaporating dish.
3. Perform a "difference weighing," i.e. — Weigh a tube containing some sand, empty some out, and reweigh the tube. Calculate how much sand was removed.

4. Check the accuracy of exercise 3 by first weighing a watch glass alone, adding the expelled sand, and weighing again.

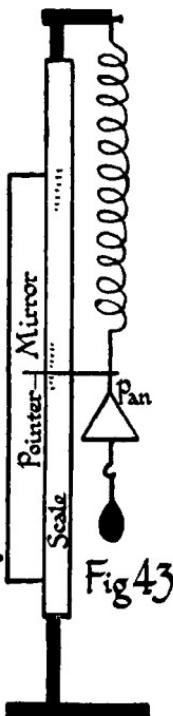
Borda devised a method for correctly weighing a substance on an incorrect balance. The substance is put on one pan of the balance, and a counterpoise on the other. This counterpoise is made of a pill-box and shot and sand. Shot is added to the empty pill-box one at a time until just too heavy; the last shot is then taken out, and grains of sand added a few at a time, until the pointer of the balance is at zero on the scale.

The substance is now removed from the pan and weights are put in its place until the counterpoise is properly balanced. Then this weight is the same as that of the original substance.

2. The Joly Balance. This instrument consists of a delicate spring suspended from a support. At the end of the spring hangs a pointer and a pan; and when required, a glass bob (Fig. 43).

The pointer moves in front of a scale attached to which is a mirror so that its position can be correctly read.

From Hooke's law we know that the elongation of the spring is (within limits) proportional to the stretching force. Therefore if a substance is put on the pan, and the reading taken, the substance removed from the pan, and weights added until the same reading is obtained, the weight of the substance is thereby obtained. When taking a reading, the pointer should exactly hide its own image in the mirror.



#### EXERCISES

- Find the elongation of the spring for a load of 1 gram.
- Find the elongation produced by adding the glass float, and hence calculate its weight.
- Weigh a watch glass with it and compare result with that obtained with an ordinary balance.
- Find the apparent loss in weight when the glass float is weighed in water and—if possible—other liquids, e.g. alcohol, sulphuric acid, glycerine, ammonia solution, zinc chloride solution.
- The Bunsen Burner. The heating apparatus for general use in the laboratory is the bunsen burner (Fig. 44), which is capable of giving two distinct flames, known as the luminous and non-luminous

according to whether the air-hole at the base is closed or open. For ordinary purposes the non-luminous flame is used.

The figure shows the correct method of heating; the hot blue zone of the flame should just reach the bottom of the substance to be heated.

4. Thermometers are used for registering temperature. A good thermometer is a necessity; ordinary boxwood and paper-backed patterns are of no use for scientific work.

They must be treated very carefully. The instruments are graduated in degrees Fahrenheit or Centigrade, the latter being generally used by scientists. As a rule the degrees are numbered at each 10

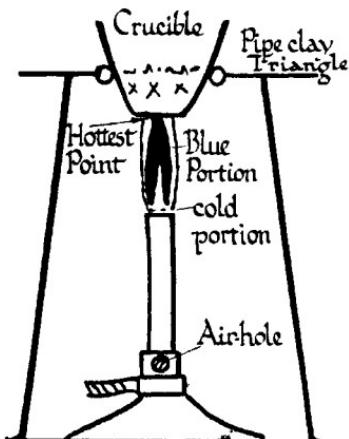


Fig 44

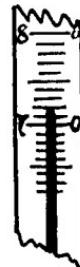


Fig 45

(Fig. 45), one figure being on each side of the graduation mark. The 5 line is longer than the others.

The relationship between the degree Fah. and the degree Cent. is shown in Fig. 46, from which it can be seen that a degree Fah. is  $\frac{5}{9}$  of a degree C.

5. Hydrometers are old-fashioned, inaccurate, and non-scientific pieces of apparatus; there are several modifications and empirical scales. They are however still frequently used in trade, although it is time they gave place to better apparatus.

Their construction is based on the principle of buoyancy or flotation. If a solid, heavier at one end, than the other, and lighter on the whole than a liquid, be placed in that liquid, it will tend to float in the liquid so that the heavier end is underneath (Fig. 47).

The heavier this end is made, the deeper the solid sinks in the liquid; and the denser the liquid, the higher a given solid floats in it.

A body of this kind, when made in a long thin form, is called a hydrometer (Fig. 48). In the top part is a paper scale of degrees, which are quite empirical. Scientific data for relative densities are always given as *Specific Gravity*—not in degrees.

Rule to convert degrees Twaddell to sp. gr. Multiply by 5, add 1,000, divide the number thus obtained by 1,000.

E.g. convert 38° Tw. to sp. gr.

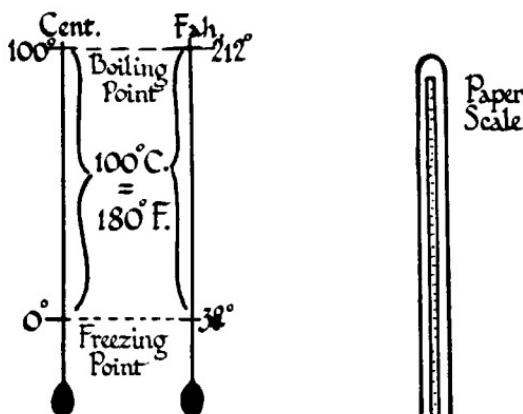


Fig 46

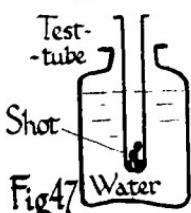


Fig 47

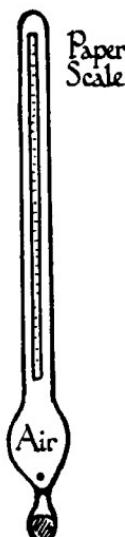


Fig 48

$$\text{Sp. gr.} = \frac{(38 \times 5) + 1,000}{1,000} = \frac{1,190}{1,000} = 1.19.$$

To convert sp. grs., reverse the process, i.e. multiply by 1,000, subtract 1,000, then divide by 5.

E.g. convert a sp. gr. of 1.76 to ° Tw.

$${}^{\circ}\text{Tw.} = \frac{(1.76 \times 1,000) - 1,000}{5} = \frac{760}{5} = 152 {}^{\circ}\text{Tw.}$$

## Beaume's Hydrometer Scales.

(a) Lighter than water.

$$\text{Sp. gr.} = \frac{144}{\text{B.} + 134} \text{ and } {}^{\circ}\text{B.} = \frac{144}{\text{sp. gr.}} - 134.$$

(b) Heavier than water.

$$\text{Sp. gr.} = \frac{144}{144 - \text{B.}} \text{ and } {}^{\circ}\text{B.} = 144 - \frac{144}{\text{sp. gr.}}.$$

*Note.*—“Twaddell” hydrometers cannot be used for liquids lighter than water. Of the many distinct Beaume scales, the above are the two best known.

## SECTION II

### I. GLASS MANIPULATION

(a) To cut glass tubing. Place the tube flat on the bench, make one cut with a triangular file; take in both hands (Fig. 49), the nick in front, and give a "pull bend."

(b) To bend glass tubing. Heat in the yellow portion of a bat's-wing or fish-tail flame (Fig. 50), holding the tube with both hands, gently rotating it all the time. The elbows should rest on the bench and the hand be held as shown in Fig. 51, which is a side view. When the glass is quite soft it should be *removed* from the flame and bent into the required shape, and held fast until it sets.



Fig 49

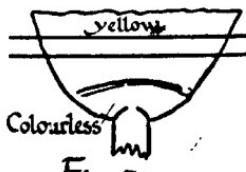


Fig 50



Fig 51

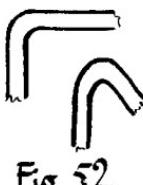


Fig 52



Fig 53

Fig. 52 shows the appearance of good glass bends. Fig. 53 shows faulty ones, due to overheating, careless bending, using wrong flame, etc.

*As an exercise make bends of the shape and dimensions given in Fig. 54 A to F.*

(c) To smooth glass ends. Use the non-luminous flame; hold as shown in Fig. 55, rotating all the time, till the glass just melts.

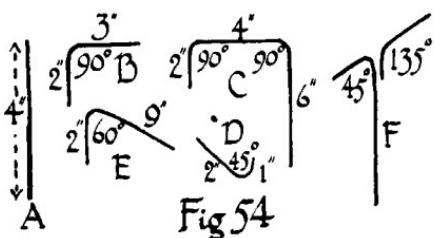


Fig 54



Fig 55

(d) To make a jet. Heat tube in a bunsen flame, remove, draw out in both directions. Cut off as shown by the dotted lines (Fig. 56), and smooth both ends.

(e) To make a closed tube or bulb tube. First make a jet, then close the end by holding it in the bunsen flame (Fig. 55). Gently blow down the open end, holding the tube vertical (Fig. 57). If the tube is to be closed only, and no bulb blown on it, the thickness should be uniform, neither sharp nor "blobbed" (Fig. 58).

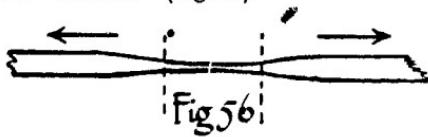


Fig 56

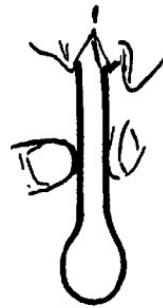


Fig 57

*Note.—Always allow hot glass to cool on an asbestos mat, and smooth all cut ends.*

## II. CORK-BORING

Select a cork slightly too large for tube, flask, etc., and roll it with gentle pressure under the foot to make it soft. Select a cork-borer slightly smaller than the tubing being used.

Hold the cork in the left hand, cork-borer in the right (Fig. 59), and bore with a screw motion, without excessive pushing. Get nearly through and then place the cork against a hard surface to finish, in

order to obtain a clean cut. Withdraw the borer by screwing in the reverse direction.

Fig. 58 shows plan and section of corks bored with one and two holes respectively. The test for a well-bored cork is the appearance of the boring—removed by pushing out with a smaller borer. This should be a perfect cylinder.



Fig. 58

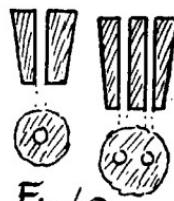


Fig. 60

Fig. 59

Practice on *waste* corks until proficiency is attained.

To insert glass tubing in a cork, first wet the tube and then screw it in with both hands close together. If pushed or forced in, the glass will break and a serious cut may result.

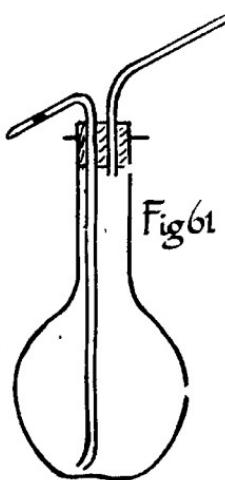


Fig. 61

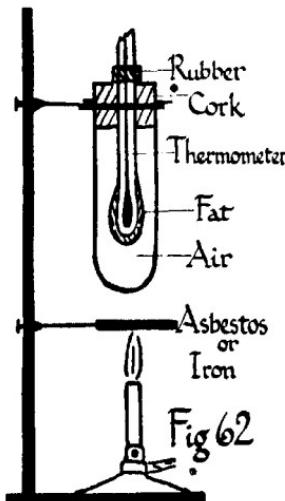


Fig. 62

### III. FITTING UP APPARATUS

- The following diagrams represent apparatus commonly used in laboratory practice, and the student is advised to set up each one in order to acquire manipulative dexterity.

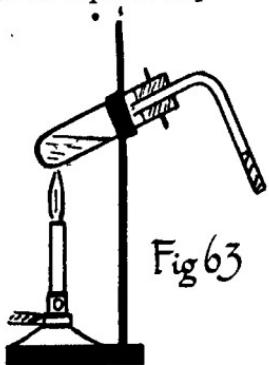


Fig 63

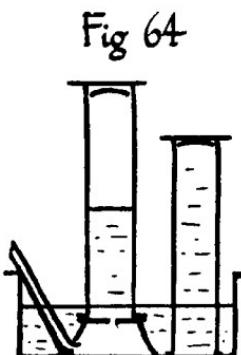


Fig 64

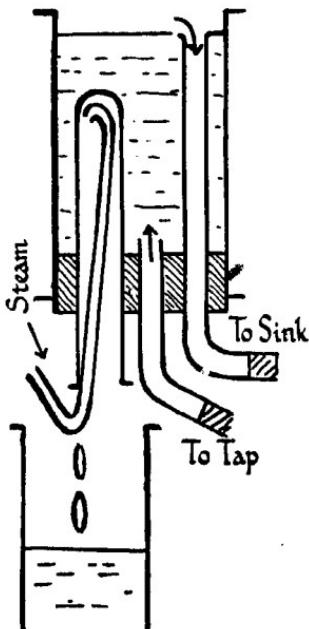


Fig 65

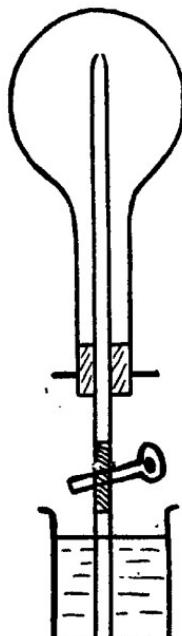
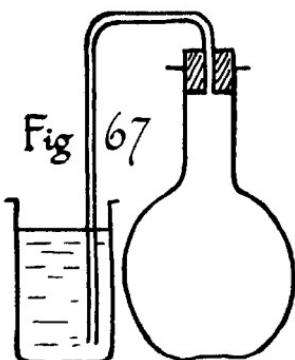


Fig 66

Fig. 61 is of the laboratory wash bottle which contains distilled water.

Fig. 62 is a simple melting-point apparatus that will be used later.

Fig. 63 represents a test tube provided with a gas leading tube, clamped on a retort stand. The evolved gas is often collected over



water. Fig. 64 shows how this is done in a pneumatic trough with a beehive shelf and gas jars.

Fig. 65 is a diagram of a simple condensing arrangement for preparation of a small quantity of distilled water.

## SECTION III

### SIMPLE PROCESSES

#### I. SOLUTION

**W**ATER is a solvent, i.e. a liquid which is capable of dissolving substances. When a substance dissolves in a liquid, it disappears to sight as a separate substance—it may however impart a colour. The substance itself is said to be soluble in the solvent and the liquid thereby produced is called a solution.

Water is not a universal solvent, but it will dissolve more substances than any other known liquid. A few substances which are insoluble, i.e. not soluble in water, are silver chloride, barium sulphate, most fats and oils. Sulphonated oils, such as Turkey red oil, are soluble in water.

Liquids which are soluble in other liquids are said to be miscible. Alcohol and glycerine are miscible with water; oils are not miscible with water as a general rule.

Gases vary considerably with respect to their solubilities in water. The most soluble gases are ammonia, hydrochloric acid, sulphur dioxide, sulphuretted hydrogen. The least soluble are hydrogen, oxygen, nitrogen, air, and carbon monoxide.

The extreme solubility of ammonia in water can be illustrated by means of the apparatus shown in Figs. 66 and 67.

As a rule the solubility of a solid substance is increased by heating the liquid (an exception is lime). Gases are expelled from solution by boiling the liquid—some completely, some only partially, as with hydrochloric acid.

Next to water the most important solvents are:—

(a) Alcohol and Methylated Spirit, which dissolves shellac, iodine, fats, resins, camphor, etc.

Solutions in alcohol are called tinctures.

(b) Carbon disulphide, which dissolves sulphur, phosphorus, fats, and oils.

(c) Ether—dissolves fats, iodine, india-rubber.

(d) Chloroform—dissolves fats, gums, resins, iodine.

(e) Benzene—dissolves fats, rubber, and many organic substances.

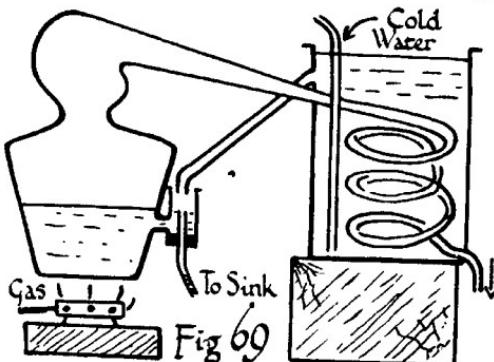
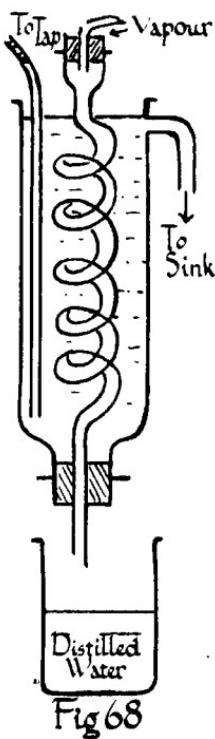
The dissolved solid can be recovered from solution by evaporating off the solvent, and the solvent can also be obtained if the product of evaporation be condensed—the process being known as distillation. The liquid which distils over is known as the distillate. Distillation may be used to prepare a compound—as nitric acid (*q.v.*, page 72 *et seq.*), or to separate two mixed liquids, such as alcohol and water.

The piece of apparatus in which the vapour is cooled is called a condenser. Fig. 22, page 6, shows a Liebig condenser, and Fig. 68 the worm form. An arrangement for preparing large quantities of distilled water is shown in Fig. 69.

Solution sometimes raises the temperature of the solvent, e.g. caustic soda in water. Sometimes the temperature falls, e.g. when ammonium chloride is dissolved. Sometimes a soluble substance is mixed with an insoluble one—solution can be used to separate them.

## II. SATURATED SOLUTION

If equal quantities of water be mixed with gradually increasing quantities of salt, it will be found that ultimately a point is reached at which the water ceases to dissolve more salt. Similar results are obtained with



other liquids and other solids. When this point is reached the solution is said to be *saturated*.

- But if a cold saturated solution be heated, more solid can be dissolved, and if this hot solution be now cooled, the excess is deposited as *crystals*.

### III. CRYSTALS

possess a definite geometric shape but have not a constant size. The size of the angles between the faces is one factor which helps to classify a crystal, which may be defined as a regular solid of definite form enclosed by four or more faces.

All crystals belong to one or other of seven systems which are distinguished one from the other by the number of planes of symmetry, and the number and inclination of the axes it is possible to obtain

from the specimen. A plane of symmetry is produced by cutting a crystal into two equal portions in such a way that one half is symmetrical in shape with the other (Fig. 70).

Crystals exhibit a property known as cleavage, i.e. a tendency to break more easily in one direction than another. Crystals may be obtained by various methods:—

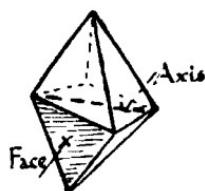


Fig 70

(1) Cooling after Fusion, e.g. prismatic sulphur, granite, and many metals.

(2) Sublimation, e.g. iodine, white arsenic.

(3) Solution and evaporation, e.g. sugar candy; salt.

(4) Cooling a hot saturated solution (see Practical Exercises, page 24).

### IV. PRACTICAL EXERCISES IN SOLUTION, ETC.

(a) To determine whether a Solid is soluble or insoluble in Water or other Liquid.

Test the solvent to see what residue it leaves on evaporation to dryness.

1. Place the substance you are experimenting with in the bottom of a test tube, half fill with the solvent, place your thumb over the end and invert several times.

2. If the solid has not disappeared, gently warm over a bunsen flame, and if necessary, boil.

3. To say if the substance has dissolved. If it has disappeared there is no doubt it has dissolved. If some solid remains, the mixture must be *filtered*.

4. Use apparatus as shown in Fig. 75, page 25. The liquid which passes through the paper into the beaker is called the *filtrate*. Note

that it is clear; if not, pass it through again. The dissolved portion is now present in the filtrate.

5. Evaporate this filtrate to dryness, using a porcelain basin on a sand bath. For more accurate work the apparatus shown in Fig. 71 is used.

6. If solid substance (other than that yielded by the evaporation of the solvent) be left in the basin the substance was soluble; if not, it was insoluble. Sometimes it is desirable to weigh the basin before and after evaporation of the filtrate, sufficient information not being obtainable by inspection.

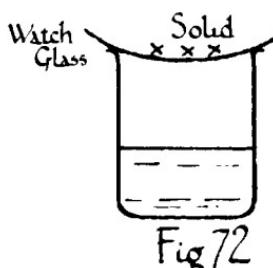
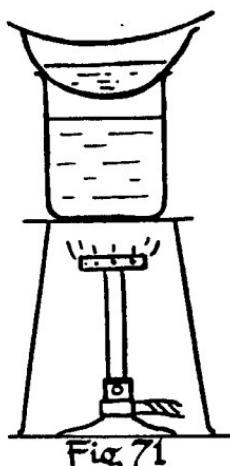


Fig. 71

Fig. 72

#### EXERCISES

Is sodium carbonate soluble in water? Is it soluble in dilute hydrochloric acid? Is sulphur soluble in water? Is tallow soluble in carbon disulphide? Is soap soluble in alcohol? Is it soluble in ether?

*Note.*—These last three solvents must not be brought near a flame.

(b) *To find if any Change in Weight occurs when a Solid dissolves in Water.*

Put a little water in a beaker, and a few crystals of ammonium chloride on a watch glass, and arrange as shown in diagram (Fig. 72). Weigh the set. Carefully tip the solid into the beaker, replace watch glass, and when the crystals have dissolved weigh the set again. What do you find?

(c) *To prepare a cold saturated Solution of a Substance and to determine the Amount of dissolved Substance in a given Volume of Solution.*

*Also to find the number of parts of Solid dissolved in 100 parts of Water.*

A. To make the cold saturated solution (say of nitre). About one-third fill a boiling-tube with water, add a teaspoonfull of nitre, and gently warm the liquid. When all the nitre is dissolved, cool under the tap. If crystals are deposited the solution is saturated. If not, more nitre must be added and the process repeated.

B. To find the dissolved solid in 100 c.c. of solution. Weigh a clean dry basin, put in 20 c.c. of solution, measured with a pipette, evaporate to expel the water, and when dry and cool, reweigh. Increase = amount of nitre in 20 c.c. Calculate for 100 c.c.

C. To find parts of solid in 100 parts of water. Weigh a dry basin, half fill with solution, and weigh again. Evaporate to dryness, weigh basin and residue. From these weighings find weight of water and weight of solid present. Calculate for 100 grams of water.

How to record weighings—

*For Exercise B.*

Basin + Residue . . . =	———
Basin only . . . . =	

Nitre dissolved in  
20 c.c. . . . . =

*For Exercise C.*

Basin + Solution . . . =	———
Basin only . . . . =	

Solution . . . . =

Basin + Residue. . . =	———
Basin only . . . . =	

Solid . . . . . =

(d) *Crystallization. To prepare Crystals of Nitre and Sal-ammoniac from Solution.*

Make a hot saturated solution of the salt. Pour into a watch glass to cool. If a thick deposit is formed, too much solid has been used. Add water and repeat. If it gets cold without forming crystals, add



Fig 73



Fig 74

more solid and repeat. When crystals are formed slowly and perfectly, make a sketch of them as shown in Figs. 73 and 74.

## (e) Preparation of Standard Solutions.

*Standard solutions* are solutions which contain a known weight of the dissolved substance in a definite volume of the solution. The usual method adopted for preparing them is to weigh out the solid and then dissolve it in *some* of the solvent. The solution thus formed is transferred without loss to a measuring vessel, and more solvent added to make up the full volume required. The vessel is stoppered, the liquid thoroughly mixed, and finally transferred to a bottle which is suitably labelled.

## EXERCISE

Prepare standard solutions containing 10 per cent. soda ash, 20 per cent. Glauber salt, and 5 per cent. common salt.

Also make a solution of the given dye stuff of such a strength that 1 c.c. of it contains .001 grams of dye.

Carefully label and preserve these standard solutions.

(f) To separate the Constituents of a Mixture of soluble and insoluble Substances, and to determine the Proportion of each present.

Transfer the mixture to a weighing bottle or test tube fitted with a cork. Weigh and record as shown below.

Empty some of the substance from the bottle into a beaker (or boiling-tube), and weigh bottle again. Enter weighing.

Add distilled water to the beaker in sufficient quantity to dissolve all the soluble portion. Stir well and warm gently. When solution is considered to be complete, filter the liquid to separate the insoluble substance from the solution.

When filtering, remember never to have the paper more than half full, and always to pour down a glass rod (Fig. 75).

Transfer every particle of insoluble substance to the filter paper by washing down with water, or (if possible) use a camel-hair mop.

Wash the substance on the filter paper, collect all washings, add to the original filtrate and evaporate to dryness in a weighed evaporating dish on a sand or, preferably, water bath or steam oven. Record weighing.

Open out the filter paper, place it on another paper from the same packet, and dry in a steam oven. Use the bottom paper to obtain weight of filter paper only.

Calculate in each case the percentage as shown on next page.

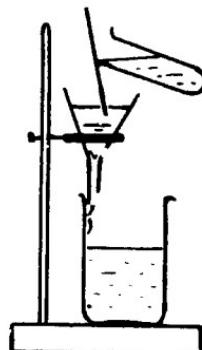


Fig 75

## TEXTILE CHEMISTRY

## METHOD OF RECORDING RESULTS

*To determine Weight of Mixture taken for use.*

Bottle + Substance at first	=	grams.
" " + Mixture left	=	"
Mixture used	=	<u><u><u>x</u></u></u> "

*2. Weight of soluble Substance present in Mixture used.*

Evaporating dish + Residue	=	grams.
" " only	=	"
Soluble substance	=	<u><u><u>y</u></u></u> "

*3. Weight of insoluble Substance present in Mixture used.*

Filter paper + Residue	=	grams.
" " only	=	"
Insoluble substance	=	<u><u><u>z</u></u></u> "

*4. To determine Percentage of each Portion.*

(a) *The soluble portion.*

Multiply the weight of soluble substance by 100 and divide by weight of mixture used :—

$$\frac{y \times 100}{x}$$

(b) *The insoluble portion.*

Multiply the weight of insoluble substance by 100 and divide by weight of mixture used :—

$$\frac{z \times 100}{x}$$

Suitable mixtures are alum and sand, nitre and sand; clay and soda ash.

As a further exercise find the proportions of the constituents in  
(1) a mixture of sand, salt, and chalk.

(2) " sulphur, nitre, and charcoal.

*Note.*—Chalk is soluble with decomposition in dilute hydrochloric acid, and sulphur is soluble without decomposition in carbon disulphide.

(3) A mixture of chalk and clay. Find a suitable solvent by experiment with separate samples of chalk and clay.

## V. FILTRATION

is the process employed for separating insoluble or suspended matter from a liquid. On a small scale, porous paper, cotton wool, or glass wool can be used, the porous substance being inserted in a piece of apparatus called a funnel.

Sand, gravel, brick ends, wood wool, charcoal, canvas, porous porcelain, and spongy iron (ignited iron oxide) are all used on larger scales of filtration, such as for a town's water supply, sugar-refining, water-softening, sewage treatment, etc.

The rate of filtration depends upon several factors, e.g.

(a) Density and nature of the precipitate or suspended matter, which may be :—

1. *Flocculent*, as copper hydroxide.
2. *Crystalline*, as lead chloride.
3. *Gelatinous*, as aluminium hydroxide.
4. *Slight* or *turbid*, as when silver chloride is precipitated from tap water.

As a rule Nos. 1 and 3 take longer to filter than Nos. 2 and 4.

(b) Temperature of the liquid. Other things being equal, a hot mixture filters much quicker than a cold one.

(c) Pressure on the surface of the liquid as it passes through the filtering medium, illustrated in the filter or suction pump.

(d) Porosity of the filtering medium. For this reason a filter paper is first wetted with clean water, so that the pores may not be partly filled up with the solid that is added first.

After a mixture has been put on a filter paper and the liquid has filtered through, the solid requires washing to remove the rest of the liquid which has adhered to the solid or the paper. This is done by blowing on it a jet of water from the wash bottle (Fig. 61, page 17).

Strong acids and caustic alkalies destroy paper; they are therefore filtered through glass wool, which is made into a tuft and placed in the funnel.

## VI. BOILING AND MELTING

A liquid is said to boil when it is in a state of active ebullition, i.e. bubbles are passing quite through its mass. The process is not identical with evaporation, and it is distinguished from it in several ways, e.g. :—

Boiling takes place at a definite temperature only—evaporation can occur at any temperature.

Boiling occurs through the whole body of the liquid—evaporation from the surface only.

Boiling is apparent to the sense of sight, but evaporation is usually invisible.

Continued boiling does not alter the temperature of the liquid (if pure); continued evaporation lowers the temperature.

These differences can be illustrated by experiments, e.g. boiling water in which is placed a thermometer; allowing alcohol and ether to evaporate on the palm of the hand; placing a dish of water in a cold room for several hours.

The temperature at which a liquid changes to a gas, when the liquid is in a state of active ebullition, is called its boiling-point (written b.p.).

Boiling-points are considerably affected by pressure. This property is made use of in the Papin digester (Fig. 76) and autoclaves, pieces of apparatus that are often employed in industrial chemistry.

The reverse process is called condensation or liquefaction.

A solid is said to melt or liquefy, or fuse, when it changes its state to the liquid condition. The melting-point (m.p.) is the temperature at which this change takes place.

Solution must not be confused with melting; e.g., in the general case sugar does not melt in water, it dissolves.

Pressure as a general rule causes the melting-point to rise. This explains why granite and other rocks are solid inside the earth, although they are at very high temperature.

In the case of water, pressure lowers the m.p. Some solids have not been liquefied yet, e.g. arsenic and carbon.

The reverse of the process is termed solidification. The boiling-point or melting-point of a substance is a very valuable aid in determining the purity of a chemical or commercial commodity, e.g. butter may be distinguished in many cases from margarine by finding the m.p. of the fatty acids in the samples.

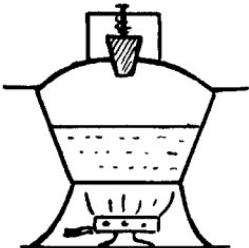


Fig 76

VII. PRACTICAL EXERCISES IN DETERMINATION OF MELTING-POINTS

Use the apparatus shown in Fig. 62, page 17, and determine the m.p. of tallow as follows. Just melt some tallow in a *small* test tube; dip the thermometer into it and remove at once. If this is done very rapidly a thin coating of fat will solidify on the bulb. Replace the coated thermometer in the boiling-tube and very slowly raise the temperature. Take the reading of the thermometer at the instant the melted fat drops from the bulb.

For obtaining accurate results the fat should be allowed to set twenty-four hours in a cold place before the test is made.

### VIII. SPECIFIC GRAVITY AND ITS DETERMINATION

By specific gravity is meant the number of times a substance is as heavy as the same volume of water. Suppose 20 c.c. of a substance weigh 18·6 grams, and 20 c.c. of water weigh 20 grams, then the sp. gr. of the substance =  $18\cdot6 \div 20 = \cdot93$ . On the face of it, it appears therefore that sp. gr. should be easily and accurately determined. In practice however it is often very difficult to determine accurately the volume of the substance.

The following methods are in general use:—

**Liquids.** *Approximate method.* Counterpoise a dry beaker, weigh in it 50 c.c. of a dilute solution of caustic soda, measured by means of a pipette (Fig. 20, page 5) or a measuring cylinder. Empty out, and weigh 50 c.c. of pure water. Calculate the sp. gr. of the solution of soda.

*Accurate method.* Carefully weigh the piece of apparatus known as a specific-gravity bottle (Fig. 77). Say when dry it weighed 12·34 grams. Fill it with the liquid whose sp. gr. it is required to determine (say a dilute solution of zinc chloride), and insert the stopper.

The excess of liquid is expelled through the perforation. Carefully wipe the bottle dry, and reweigh, say = 72·63 gr. Then weight of this volume of liquid =  $72\cdot63 - 12\cdot34 = 60\cdot29$  grams.

Empty out the liquid, clean the bottle, and fill it in the same way with pure water. Wipe and weigh again, say = 62·21 grams. Then the weight of this volume of water =  $62\cdot21 - 12\cdot34 = 49\cdot87$  grams.

Then, as the volumes are the same, the

$$\text{Sp. gr.} = 60\cdot29 \div 49\cdot87 = 1\cdot21.$$

*Commercial method,* using hydrometers (Fig. 48, page 13). The method of using these instruments is given on pages 12–14. The liquid must be put in a gas jar about  $1\frac{1}{2}$  inches wide, so that the hydrometer can float freely. The reading is taken at the surface of the liquid (Fig. 78).

As an exercise find the sp. gr. of glycerine, strong caustic soda solution, methylated spirit, concentrated sulphuric acid, concentrated hydrochloric acid, ammonia solution, and pure distilled water.

*Rapid accurate method.* One of the quickest and most accurate



Fig 77

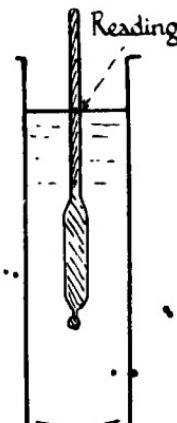


Fig 78

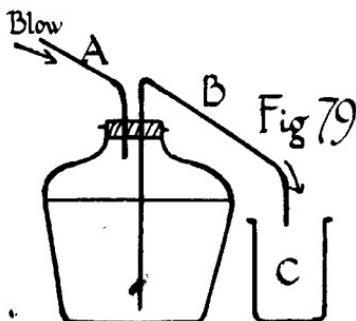
methods of finding sp. grs. of liquids is by the use of the Joly balance (Fig. 43, page 11). Remove the pan of the balance and substitute the glass bob. Take the reading of the pointer when the bob is floating in air—say graduation mark is 163. Partly fill a beaker or large test tube with water, bring it underneath the bob, and allow the latter to be freely and completely immersed. Take reading again, say = 147. Then the *upward push of the water* is represented by  $163 - 147 = 16$  graduations.

Remove vessel of water, carefully wipe the bob, and repeat with the liquid whose sp. gr. it is required to find.

Say reading is 149. Then *upthrust of the liquid* is represented by  $163 - 149 = 14$  graduations.

$$\text{Then sp. gr.} = \frac{\text{upthrust of liquid}}{\text{upthrust of water}} = \frac{14}{16} = .875.$$

As an exercise use the same liquids as with hydrometers, and compare the results.



Liquid chemicals are often supplied to mills and works in carboys, drums, or similar vessels, e.g. strong acids, alkalies, zinc and magnesium chlorides, oils, etc. The safest and simplest method for removing samples for testing is to fit up an arrangement, similar to that adopted for the laboratory wash bottle (Fig. 61, page 17). A good and tight-fitting cork carrying two glass tubes should be inserted into the mouth of the carboy (Fig. 79). By blowing at the end of tube A, liquid is driven out through tube B and can be collected in vessel C.

**Solids.** The best way to find sp. gr. of fats and waxes is to make very small pellets of them, and mix alcohol (either ethyl or methyl will do, but ordinary methylated spirit is not suitable, due to precipitation of gum) and water till the pellets will neither sink nor float in the mixture except under compulsion. Then find the sp. gr. of this liquid, which is identical with the sp. gr. of the solid.

## EXERCISE

Find sp. gr. of tallow, spermaceti wax, and paraffin wax.

## IX. EFFECTS OF HEAT

The general effect of heat upon a substance is to produce a change. Sometimes this change is physical, sometimes chemical, sometimes both (see Section IV).

Under the head of physical there are :—

- (a) Alteration in volume, usually an increase.
- (b) Decrease in density.
- (c) Change in state, e.g. solid to liquid.

Among chemical effects produced are found :—

- (a) Decomposition of compounds.
- (b) The dehydration of substances containing moisture and water of crystallization.
- (c) The formation of chemical compounds from constituent elements.
- (d) The decomposition of original compounds and formation of new ones.
- (e) The conversion of a mixture of substances into one or more compounds.

The process of strongly heating a solid is often termed *ignition*—it is not identical with the ordinary use of the word.

## EXERCISES

1. Heat mercuric oxide in a test tube. Test gas evolved with a glowing splint.
2. Perform a similar experiment with potassium chlorate.
3. Heat crystals of bluestone or copper sulphate.
4. Heat magnesium ribbon in air.
5. Heat wood in a test tube and note inflammable gas produced.
6. Heat a mixture of iodine and mercury in a test tube.

## X. DETERMINATION OF PERCENTAGE OF WATER IN SUBSTANCES

Many raw materials and substances used in the textile industry contain water, the proportion of which it is often desirable to know, e.g. cotton yarn and cloth, starches, fats, soaps, "compositions," solutions of caustic soda, zinc chloride, magnesium chloride, etc.

If the substance is a solid it is weighed in a wide test tube or weighing bottle (Fig. 80) or aluminium tray, or pair of watch glasses clipped together (Fig. 81), or glass evaporating-basin; and then put into a steam oven until there is no further loss in weight. The total loss

represents moisture (and other constituents volatile at 100° C.). It is returned as a percentage.

If the substance is a liquid, it should be put into a small test tube 3 in.  $\times$   $\frac{1}{4}$  in., made to stand upright by placing the end in a cork (Fig. 82). The method of working is exactly the same as that for a solid.

#### EXERCISES

Find percentage of water in flour, farina, soap, caustic soda solution, zinc chloride solution, magnesium chloride solution.

#### XI. TO DETERMINE PERCENTAGE OF ASH

The substance is first weighed in a crucible, then heated over the bunsen flame, at first gently, then strongly until a grey white residue is

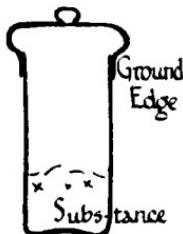


Fig 80

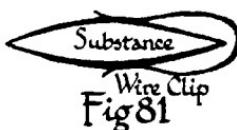


Fig 81

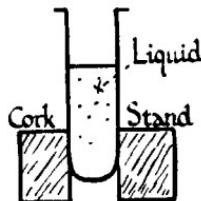


Fig 82

obtained. The crucible lid must be removed while the heating is in progress, and the flame must be arranged as shown in Fig. 44, page 12. The hot blue zone of the flame should just reach the bottom of the crucible.

Many textile materials greatly increase in volume and sometimes froth on heating; hence the crucible should never be more than one-third full; and it is generally advisable to remove the burner for a few minutes at intervals.

As a rule a black mass is obtained first. If it is hollow it should be broken gently *when cold* and made to fall to the bottom of the crucible. This blackness is due to the presence of carbon. The heating, which may now be intense, must be continued until all black is burnt away, as *carbon is not ash*. The whiter the residue obtained the better will be the result.

Results should be tabulated in the following way :—

Weight of crucible and lid + Substance =

“	”	”	only	$\overline{\overline{=}}$
---	---	---	------	---------------------------

$\therefore$  Amount of substance used =  $\underline{\underline{a}} \text{ gm.}$

$$\begin{array}{lcl}
 \text{Weight of crucible and lid + Ash} & = & \\
 " " " " \text{ only} & = & \hline \\
 \therefore \text{Amount of ash} & = & \underline{\underline{b}} \text{ gm.}
 \end{array}$$

$$\text{Then percentage ash} = \frac{b \times 100}{a}$$

## EXERCISES

Determine the percentage of ash in powdered magnesite, flour, commercial glycerine, sago flour.

An exercise which combines a moisture and ash determination is one with China clay, to find percentage of "free" and "combined" moisture.

First dry some in a steam oven and find loss per cent. This is "free moisture."

Next take this steam-dried clay, and heat over a bunsen burner as in ash determinations, until there is no further loss. This second loss gives the "combined moisture."

Record results as follows:—

$$\begin{array}{lcl}
 \text{Clay + crucible} & = & \\
 " \text{ only} & = & \hline \\
 \therefore \text{Clay} & = & \underline{\underline{5.38}} \text{ (say) gm.}
 \end{array}$$

$$\begin{array}{lcl}
 \text{Crucible + Clay not dried} & = & \\
 " + " \text{ steam-dried} & = & \hline \\
 \therefore \text{Free moisture} & = & \underline{\underline{.1}} \text{ (say) gm.}
 \end{array}$$

$$\begin{array}{lcl}
 \text{Crucible + Clay steam-dried} & = & \\
 " + " \text{ ignited} & = & \hline \\
 \therefore \text{Combined moisture} & = & \underline{\underline{.613}} \text{ (say) gm.}
 \end{array}$$

$$\text{Percentage free} = \frac{.1 \times 100}{5.38} = 1.86 \text{ per cent.}$$

$$\text{Percentage combined} = \frac{.613 \times 100}{5.38} = 11.4 \text{ per cent.}$$

N.B.—Both results are calculated on the original weight of clay, i.e. 5.38 gm.

## SECTION IV

### I. CLASSIFICATION OF MATTER

**T**HE word *matter* is used to include practically an infinite number of things—hence the need for classification.

For physical purposes there are two classes : (1) Solids. (2) Fluids subdivided into liquids and gases. The chemist has adopted other classifications, some of which are not perfect—the classes overlap or are not inclusive.

The most important chemical classification is the one which divides matter into (1) Elements, (2) Compounds, (3) Mixtures ; and every known substance can be placed in one or other of these three classes.

An Element is a thing which has not by any known means been resolved into anything simpler—it yields nothing but itself. Ninety substances are known which have not been split up. Some of the commonest are : carbon, iron, copper, sulphur, lead, silver, gold, oxygen, nitrogen, hydrogen, aluminium, phosphorus, mercury, iodine, sodium, potassium, chlorine, tin, magnesium, zinc.

Elements are divided into two sub-classes : (a) Metals, (b) Non-metals. This however is not a perfect division, as there are some elements which seem to belong strictly to neither class. These are called metalloids. Arsenic is one example.

Metals as a rule possess the following characteristic properties : (1) are malleable, (2) are ductile, (3) possess a peculiar lustre, (4) have a high specific gravity, (5) ring when struck, (6) are good conductors of heat and electricity.

They also possess the property of intimately mixing with each other when melted together or compressed, to form *alloys*. Some common alloys are pewter, bronze, German silver, type metal, solder, fusible metal.

Non-metals are elements which do not possess metallic properties, i.e. they are not malleable, not ductile, etc. This class includes all the elementary gases, and carbon, sulphur, phosphorus, and iodine.

Compounds are things which contain *two or more elements* united together in certain *definite proportions* in the smallest piece of the

substance which is capable of having an existence as such. A compound *must* be homogeneous in structure.

Compounds are very numerous—over half a million are known. Some common ones are : water, salt, washing soda, sand, alum, sugar, ammonia, copper sulphate, starch, China clay, glycerine, copper oxide, zinc chloride, magnesium chloride, chalk.

Mixtures are the most commonly occurring of all things. In these there is no definite structure in their smallest particles. Examples —milk, coal, tar, putty, flour, wood, glass, gunpowder, granite, fibres, air, soap, and most oils and fats.

There is no invariable rule by which we can distinguish a pure substance (i.e. an element or compound) from a mixture. Sometimes the distinction is difficult to make, but as a rule it is fairly easy. The following are some of the principal differences between mixtures and pure substances in the solid form :—

#### MIXTURES

1. Constituents can be distinguished by the eye, with or without the aid of a microscope.
2. By solution and crystallization crystals of different kinds may be obtained.
3. In many cases a particular solvent dissolves part of the mixture, leaving an insoluble residue.
4. They have no definite boiling or melting points.

#### PURE SUBSTANCES

1. The appearance is uniform throughout, however minutely they are examined.
2. Solution and crystallization give crystals of one kind only.
3. Substance dissolves thoroughly and uniformly.
4. Exhibit invariable and definite melting and boiling points.

### II: SIMPLE TESTS FOR IDENTIFICATION OF COMMON SUBSTANCES used in trade or laboratory

#### (a) Black Substances

*Manganese dioxide.* Amorphous powder ; heated with hydrochloric acid, yields a green gas of irritating odour called chlorine.

*Copper oxide.* Amorphous ; heated in contact with hydrogen or coal gas, is reduced to metallic copper ; gently warmed with dilute sulphuric acid, is dissolved to form a blue solution of copper sulphate.

*Charcoal.* Insoluble in acids ; burns in oxygen or air to form carbon dioxide, which turns lime water milky.

#### (b) Metallic Substances

*Copper.* Soft red metal ; soluble in nitric acid to a green solution.

*Zinc.* Hard and white ; soluble in hydrochloric acid with evolution of hydrogen gas.

*Lead.* Soft ; cut by a knife ; rapidly tarnishes ; insoluble in dilute hydrochloric and sulphuric acids.

*Magnesium.* Burns with bright white flame ; very soluble in dilute acids.

*Aluminium.* Very light ; silver white ; soluble in caustic soda solution with evolution of hydrogen.

*Iron.* Grey ; soluble in dilute sulphuric acid to a green solution with evolution of hydrogen ; addition of nitric acid to this solution produces a darker and sometimes a black colour.

#### (c) Coloured Substances

*Ferrous sulphate.* Green crystals soluble in water ; if ammonia is added to this solution a blue precipitate is produced.

*Copper sulphate.* Blue crystals soluble in water ; if a bright knife is immersed in this it becomes plated with copper.

*Mercuric oxide.* Orange yellow or red powder ; when heated it darkens in colour, gives off oxygen, and forms a mirror of mercury higher up the tube.

*Red lead.* Bright red powder ; heated, it evolves oxygen and turns to a straw colour ; gently warmed with dilute nitric acid, some dark brown lead peroxide is produced.

*Lead peroxide.* Dark brown ; gently warmed with hydrochloric acid, it evolves chlorine.

*Sulphur.* Yellow crystals ; soluble in carbon disulphide ; burns with a blue flame.

#### (d) White Substances

*Potassium chlorate.* When heated decrepitates, melts, effervesces, gives off oxygen, and leaves a white residue.

*Potassium nitrate.* For shape of crystals, see Fig. 73, page 24 ; heated with strong sulphuric acid, brown fumes of nitric acid are evolved.

*Ammonium chloride.* Heated with caustic soda, ammonia is evolved ; for shape of crystals on recrystallization, see Fig. 74, page 24.

*Marble.* Dissolves in acid, liberating carbon dioxide, which turns lime water milky.

*Alum.* Soluble in water, giving an astringent taste ; yields a white gelatinous precipitate on adding ammonia.

*Salt.* Cubical crystals soluble in water ; insoluble in strong hydrochloric acid.

*Sugar (cane).* Cubical crystals ; sweet taste ; soluble in water ; melts to a dark brown liquid ; strong sulphuric acid carbonizes it.

*Glucose (commercial).* Yellow to brown solid ; not so sweet as cane

sugar ; boiled with Fehling Solution, a bright red precipitate of cuprous oxide is formed.

*Sodium carbonate.* Caustic taste ; soapy feel ; turns red litmus blue ; if as washing soda, it effloresces in air.

*Starch.* Boiled with water, cooled, neutralized with acetic acid (if alkaline) and then tincture of iodine added, a blue colour is obtained.

*Dextrine.* Soluble in cold water ; tincture of iodine added to this solution, a violet colour is obtained with the commercial variety.

*Bleaching powder.* Treated with dilute acid, chlorine is evolved ; very deliquescent as usually met with.

*Caustic soda.* Very hygroscopic in air ; forms strongly alkaline solution ; gives intense yellow colour to bunsen flame.

*Flour.* Gives the reaction for starch, and is also turned yellow by strong nitric acid.

### III. PHYSICAL AND CHEMICAL CHANGES

There is no such thing as constancy in nature—evolution and change are always taking place, in fact chemistry has sometimes been called the science of change.

Rubbing a needle with a magnet will produce a change, as will rubbing a match on a piece of sand-paper.

Mixing sugar and water causes the disappearance to sight of the sugar ; mixing sugar and water with strong sulphuric acid changes the first-named to carbon.

Warming sulphur, or iodine, or alcohol changes the state of these bodies, and exposure of iron to the atmosphere changes it to rust.

Careful examination of these and other examples shows us that all changes can be classified under one or other of two heads :—

(a) **Physical change**, in which the intimate constitution of the substance is not affected—such as ice into water, water into steam, boiling of alcohol, ether, etc., solution of sugar in water.

(b) **Chemical change**, in which there is an alteration in composition, the original substance being transformed into something else, e.g. action of sulphuric acid on sugar, action of heat on potassium chlorate, mercuric oxide, etc.

Be careful *not* to define a chemical change as one brought about by the action of heat. All the various agencies are capable of bringing about both kinds of change.

The following experiments result in the production of *typical chemical changes* :—

1. Heat potassium chlorate in a test tube, gas given off which reignites a glowing splint ; white substance left.
2. Heat mercuric oxide in a test tube, turns brown in colour ; gas

given off which reignites a glowing splint ; globules of mercury sublime higher up the tube.

3. Heat mercury and iodine in a test tube, violet fumes ; then yellow, and ultimately a red sublimate formed.

4. Rub iodine and mercury in a mortar ; first a green, then yellow, then red substance formed.

5. Expose bright sodium to air, tarnished on the surface.

6. Mix hydrochloric acid and ammonia gases, a solid in the form of white fumes is produced.

7. Submit silver chloride to the action of light, it changes in colour.

#### IV. HOW TO TELL IF A CHEMICAL CHANGE HAS TAKEN PLACE

Look for :—

1. Change in texture, examining with a lens if necessary.
2. Evolution or not of gases.
3. Changes in solubility, boiling-point, melting-point.
4. Alteration in mass or volume.
5. Conduct with various reagents.
6. Evolution of heat or otherwise.
7. Action with well-known solvents, e.g. water, alcohol, carbon disulphide.

#### V. EXAMPLES OF PHYSICAL AND CHEMICAL CHANGES produced by various agencies

##### HEAT

1. Ice changed to water, water to steam.
2. Volatilization of mercury, iodine, sulphur, etc.  
(Physical changes.)
3. Decomposition of potassium chlorate, mercuric oxide, ammonium chloride, etc.
4. Combination of iron and sulphur, copper and sulphur, lead and oxygen.  
(Chemical changes.)

##### ELECTRICITY

1. Current heating a wire or filament through which it travels, e.g. electric light.  
(Physical change.)
2. The "sparking" of gases.  
Hydrogen and oxygen combine to form water.  
Carbon monoxide and oxygen combine to form carbon dioxide.  
Ammonia gas is resolved into hydrogen and nitrogen.

3. Decomposition of liquids and solids by a current in the process of electrolysis.

Water into hydrogen and oxygen.

Copper deposited from solutions of copper sulphate.

Molten caustic soda or potash to produce the metal.

(Chemical changes.)

#### FRICTION

1. Melting ice by rubbing pieces together.  
(Physical change.)
2. Rubbing the head of a match on a rough surface.
3. Rubbing iodine and mercury together in a mortar to produce mercuric iodide.  
(Chemical changes.)

#### EXPOSURE TO THE ATMOSPHERE

1. Action of freezing water on rocks and soils.
2. Absorption of water by strong sulphuric acid, caustic soda.  
(Probably physical changes.)
3. Phosphorus, iron, etc., oxidize in air, silver combines with sulphur.
4. Phenomena of tarnishing, rusting, and fermentation generally.  
(Chemical changes.)

## SECTION V

### WATER

**W**ATER is one of the fundamentals of existence as we know it. It comprises three-quarters of the earth's crust and is present in most natural things, in many of them to a very high degree, e.g. fish 80 per cent., animals (including man) up to 70 per cent., plants 50 to 95 per cent., and even in clay up to 14 per cent.

With one exception all the natural sources of water are more or less impure. The pure form is water vapour, which is always found in the

atmosphere. From *all* surfaces of water exposed to the air evaporation is always taking place, and the water vapour so formed rises (because it is lighter than air, 0·62 : 1). It is thus cooled, and in the end condensed and falls as rain, etc.

Rain-water in its fall dissolves gases from the atmosphere (oxygen, carbon dioxide, and nitrogen). This can be demonstrated by arranging the experiment shown in Fig. 83. When the water is heated, the gas is expelled and led by means of the funnel to the collecting tube.

An average sample of rain-water will yield about 25 c.c. of gas per litre, of which over 30 per cent. will be oxygen, and nearly 3 per cent. carbon dioxide.

Rain-water falling through a town's atmosphere will contain solid matter also.

When the rain reaches the ground some soaks in (25 to 40 per cent.) and some runs along the surface, forming rivers and springs. This water contains solids as well as gases. They may be present in two forms :—

1. *Suspended solids.* These can be seen and may be removed by the process of filtration (Section III, page 27). Many of them are of

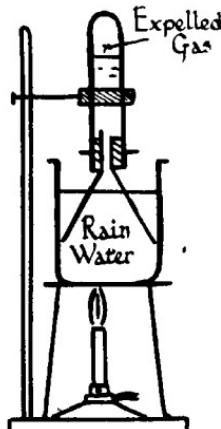


Fig. 83

an organic character, e.g. canals and most rivers flowing through large towns are used for trade effluents which often contain a large amount of suspended matter.

**2. Dissolved solids.** These can be removed by the process of distillation only (Section III, page 21). The nature of the dissolved solid will depend upon the kind of strata over, or through which, the river or spring has passed, e.g.:-

(a) Chalk or calcium carbonate in the Eastern and Southern Counties of England.

(b) Sulphate of lime in the River Trent.

(c) Magnesium salts at Epsom and in Durham.

(d) Peaty matter on the Lancashire moors.

(e) Chlorides of sodium and potassium in sewage effluents.

The amount of solids found in water may vary from .032 gram per litre in Loch Katrine to 230 grams per litre in the Dead Sea. Dissolved solids alter the properties of water, particularly in reference to its action on soap. Pure water, when mixed with soap, forms a lather and is said to be *soft*, but waters containing lime or magnesium salts prevent the soap forming a lather readily, and are said to be *hard*. (See practical exercises, pages 43 and 44.)

The hardness of and total solids in water are very important factors for consideration when deciding if a water is suitable for trade purposes. A water showing 5 degrees of hardness is termed soft, up to 18 to 20 degrees moderately hard, and over 30 degrees very hard.

Very soft water is liable to dissolve metals such as lead, zinc, iron, etc., from pipes and storage vessels; so for domestic purposes this water is sometimes "hardened" by the addition of lime.

It is also usual to soften hard water by the use of lime or washing soda, or caustic soda or other chemicals. The rationale of the process where lime is used is as follows: The "hardening salts" are present as bicarbonates of calcium or magnesium, which are *soluble* in water. The addition of lime converts them into the normal carbonates, which are nearly *insoluble* in water, with the result that they are precipitated. If this precipitate be allowed to settle, the clear softer water can be withdrawn. This is known as Clarke's process for water-softening.

Sea water contains a large quantity of sodium chloride in addition to lime and magnesium salts. The presence of this substance is demonstrated by adding a solution of silver nitrate, when a heavy white precipitate is obtained.

Pure water can be obtained from the natural sources by the process of distillation (Fig. 69, page 21).

It should be (a) tasteless; (b) colourless; (c) odourless; (d) completely volatile; (e) neutral to litmus and lacmoid; (f) able to produce a lather when 50 c.c. of it are shaken up with 1 c.c. of standard soap

solution. It has a b.p. of  $100^{\circ}\text{C}$ . or  $212^{\circ}\text{F}$ ., and is a bad conductor of heat and electricity.

As an exercise in the preparation of pure water, fit up the apparatus shown in Figs. 84, 85, 86. Reject the first portion which comes over,

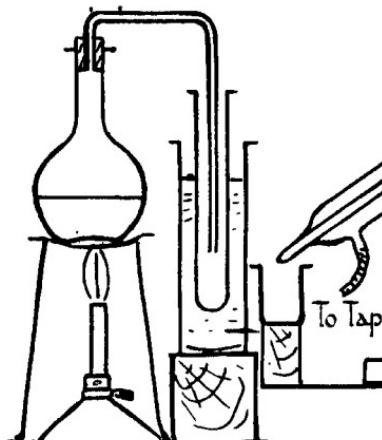


Fig 84

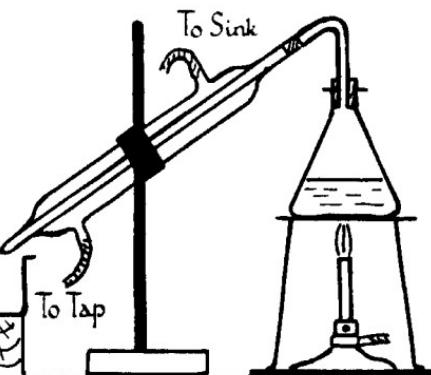


Fig 85

as this might have dissolved some matter in passing through the glass tubes. When obtained, perform the following experiments :—

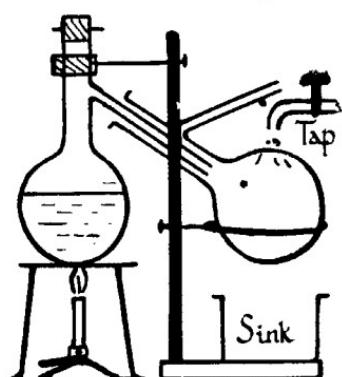


Fig 86

(a) Evaporate some to dryness ; evaporate the same quantity of well water or spring water, canal water or tap water, and compare the residues by inspection and by weighing.

(b) Taste it, and compare the taste with tap water.

(c) Note its action on litmus and lacmoid.

(d) Find how many drops of standard soap solution are required to produce a permanent lather with 10 c.c. of it. Compare with the amount required by 10 c.c. of tap water. Shake well after adding each drop, use a test tube and close tightly with the thumb.

(e) Add a few drops of silver nitrate solution. What occurs ? Compare with tap and canal water. •

A simple analysis of a natural water to determine suitability for textile purposes should include the following :—

1. Determination of *total solids*.

Weigh a clean dry beaker, put in 200 c.c. of the water to be tested, and keep in a steam oven until evaporated to complete dryness. Weigh again. Increase = Total solids in 200 c.c. Calculate to 100,000, i.e.  $\times$  by 500. After weighing, dissolve the residue in a few drops of dilute hydrochloric acid.

Note if *carbonates* are present—effervescence.

Test for *sulphates* by adding to a few drops of the liquid some barium nitrate solution. White precipitate if present.

Test for *lime*. To another portion of the solution, add ammonia, ammonium chloride, and ammonium oxalate solution. White precipitate = Lime.

2. Determination of *total hardness*.

Take 50 c.c. of water in a bottle provided with a tight-fitting cork. Put "standard soap" solution in a burette (Fig. 21, page 5), and run it into the water a *few drops* at a time, shaking well after each addition. Repeat until a permanent lather is produced which lasts without breaking for three minutes when the bottle is laid on its side, and is about  $\frac{1}{2}$  inch thick. Perform the experiment three times, find the average amount of soap solution used, and by consulting the Table of Hardness (page 44) find the degree of hardness of the water.

3. To determine *permanent hardness*.

(Permanent hardness is due to the presence of hardening salts, which are not removed by boiling the water.) Take 250 c.c. of the water and boil gently for half an hour. Filter quickly, and when cold make up to the original volume with distilled water and mix thoroughly. Find the amount of soap required by 50 c.c. as for total hardness, and obtain result in a similar manner.

4. *Temporary hardness*.

Obtained by calculation.

Total — Permanent = Temporary.

5. To test for *metals*.

Put some water in a white porcelain dish, stir with a glass rod which has been dipped in ammonium sulphide. Blackening = iron, lead, or copper. Add a few drops of acetic acid. Colour disappears = iron; colour remains = lead, or copper, or both.

## TEXTILE CHEMISTRY.

## TABLE OF HARDNESS

(Hardness of water expressed as grams of calcium carbonate per 100,000 c.c. of water.)

c.c. Soap.	Gm. CaCO <sub>3</sub> .	c.c. Soap.	Gm. CaCO <sub>3</sub> .	c.c. Soap.	Gm. CaCO <sub>3</sub> .
1·0	.48	6·0	7·43	11·0	14·84
1·5	1·27	6·5	8·14	11·5	15·63
2·0	1·95	7·0	8·86	12·0	16·43
2·5	2·6	7·5	9·57	12·5	17·22
3·0	3·25	8·0	10·3	13·0	18·02
3·5	3·9	8·5	11·05	13·5	18·81
4·0	4·57	9·0	11·8	14·0	19·6
4·5	5·29	9·5	12·56	14·5	20·4
5·0	6·0	10·0	13·3	15·0	21·19
5·5	6·71	10·5	14·06	15·5	22·02

For volumes of soap solution used between those given in the table estimate the grams of calcium carbonate, e.g. 3·2 c.c. = 3·51.

If more than 15·5 c.c. of soap solution are required, 25 c.c. of the water being tested should be used, 25 c.c. of distilled water added, and the hardness so obtained multiplied by two.

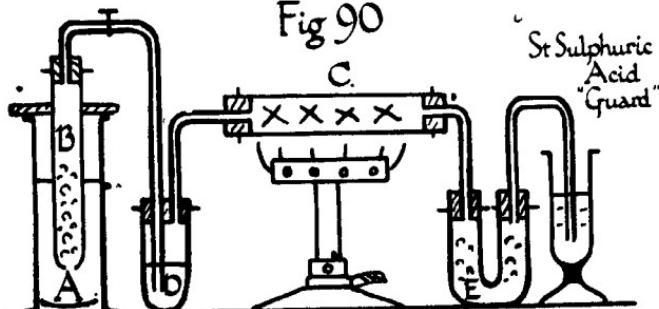
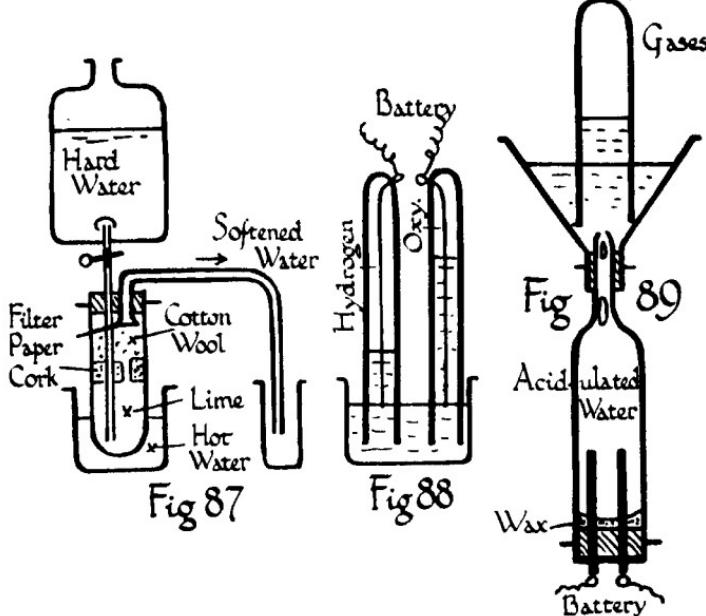
*Standard soap solution* is prepared by dissolving pure soap in a mixture of ethyl alcohol (2 vols.) and distilled water (1 vol.), allowing the mixture to stand a few days, decanting the clear liquid and setting this against a "standard hard water" until it is of such a strength that 14·2 c.c. of soap solution are required to form a permanent lather with 50 c.c. of standard hard water.

*Standard hard water* is made by dissolving 0·4 grams of pure marble in hydrochloric acid, driving off the excess of acid, and dissolving in 2 litres of pure water. This water has a hardness equal to 20 grams of calcium carbonate per 100,000 c.c. of water.

The principle of the construction of a continuous water-softening plant can be illustrated by arranging the apparatus as shown in Fig. 87. Hard water is placed in the aspirator and allowed to flow to the bottom of a large test tube which is divided into two compartments by a perforated cork. In the bottom is put milk of lime, and in the top is packed cotton wool. The exit pipe is covered with filter paper. The hard water is softened when in contact with lime, passes through the cork, and the precipitated chalk and excess of lime is filtered out in its passage upwards, with the result that a much softer water is collected in the beaker. The bottom of the test tube may be surrounded with a vessel containing warm water, when the efficiency is increased.

With a piece of apparatus arranged as shown, water requiring 16 c.c. of standard soap solution per 20 c.c. was softened to one requiring only 4 c.c.

When a fairly strong current of electricity is passed through *acidulated* water, it is decomposed into hydrogen (2 vols.) and oxygen (1 vol.) (Fig. 88). Hydrogen is liberated where the current leaves, and oxygen where the current enters the liquid. If the gases be collected together



(Fig. 89), and the mixture sparked, they recombine with explosive violence to form water.

The synthesis, or building together, of water, may be studied by using the apparatus illustrated in Fig. 90.

Hydrogen is generated from zinc (B) and dilute sulphuric acid (A) dried by bubbling through strong sulphuric acid (D) and then passed over heated copper oxide contained in a hard glass tube (C). The copper oxide is reduced, the oxygen combining with the hydrogen to form water, which is collected in the U tube E, containing an absorption agent such as calcium chloride.

Results obtained in an actual experiment with this apparatus :—

$$\text{Wt. of tube + Copper oxide before heating} = 42.179 \text{ gm.}$$

$$\text{“ “ + “ “ after “ “} = 42.150 \text{ “}$$

$$\therefore \text{Oxygen used} = \underline{\underline{.029}} \text{ “}$$

$$\text{Wt. of calc. chlor. and tube after absorption} = 41.653 \text{ gm.}$$

$$\text{“ “ “ before “ “} = 41.620 \text{ “}$$

$$\therefore \text{Water produced} = \underline{\underline{.033}} \text{ “}$$

$$\text{Hydrogen used} = .033 - .029 = .004 \text{ gm.}$$

$$\text{Ratio} = 1 : 7.25.$$

*Chief uses for water—*

1. Domestic purposes—washing, cleansing, food, drink.
2. To generate steam for motive and other purposes.
3. For motive purposes—water mills, turbines, etc.
4. Irrigation, navigation, solution, etc.
5. Water vapour forms an “earth blanket.”

SECTION VI

### I. GASES

**A** GAS has neither definite shape nor size ; it possesses the property of *expansibility*. Some gases are light, some heavy, some coloured, some invisible, some elements, some compounds, some mixtures, some soluble in water, some nearly insoluble, but certain properties they have in common.

Name of Gas.	Relative Density.	Class.	Solv. in Water.	Smell, Colour, etc.
Hydrogen . . . .	1	Element	Insol.	No smell
Coal gas . . . .	4	Mixture	"	Smell due to impurities
Marsh gas . . . .	8	Compound	Vy. sol.	No smell
Ammonia . . . .	8.5	C.	Nearly insol.	Pungent
Nitrogen . . . .	14	E.	{ Insol.	No smell
Carbon monoxide .	14	C.	{ Insol.	
Air . . . .	14.4	M.	Slig. sol.	Brown fumes in air
Nitric oxide . . . .	15	C.	Insol.	No smell
Oxygen . . . .	16	E.	Slig. sol.	Fetid smell
Hydrogen sulphide .	17	C.	Sol.	White fumes in air
Hydrochloric acid .	18.2	C.	Vy. sol.	Faint acid smell
Carbon dioxide . .	22	C.	Sol.	Laughing gas
Nitrous oxide . . .	22	C.	"	Suffocating
Sulphur dioxide . .	32	C.	"	Green colour
Chlorine . . . .	35.5	E.	"	Brown colour
Bromine . . . .	40	E.	"	

To find the weight of a given volume of gas, exhaust a globe and counterpoise it (Fig. 91). Fill with gas and reweigh. Find its volume by measurement of diameter and calculation. Volume of sphere =  $\frac{4}{3} \times \frac{22}{7} \times r^3$ .

To find the solubility of a gas, fit up the apparatus as shown in Fig. 92 (or Figs. 66, 67, pages 18, 19). The graduated tube is filled with the gas over mercury and the solvent is put in the cup.

When the tap is cautiously opened, a few drops will fall into the tube and float on the mercury. The tap is closed, and the liquid

allowed to exert its solvent action on the gas. As it does so the mercury rises in the tube.

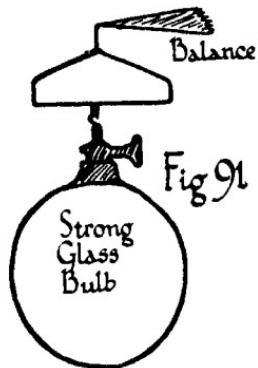
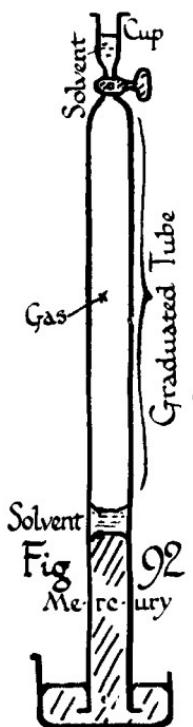


Fig 91



Some general methods for preparation of gases—

1. *Boil liquids.* Water, ether, alcohol, carbon disulphide.
2. *Heat solids.* Wood, coal, iodine, chlorate of potash.
3. *Treat solids with acids.*  
Copper and nitric acid—brownish red gas.  
Copper and sulphuric acid—sulphur dioxide.  
Zinc and hydrochloric acid—hydrogen.  
Marble and hydrochloric acid—carbon dioxide.  
Salt and sulphuric acid—hydrochloric acid.  
Manganese dioxide and hydrochloric acid—chlorine.

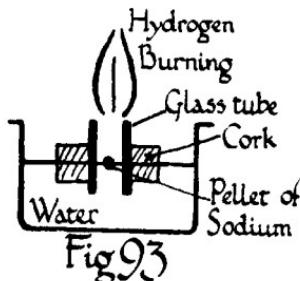


Fig 93

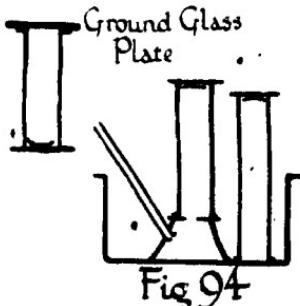


Fig 94

4. *Treating a liquid with a solid.* Water and sodium or potassium give hydrogen (Fig. 93).

5. *From a mixture of two gases remove one.* Burn phosphorus in air—nitrogen is left (Fig. 107, page 54).

6. *Electrolysis of liquids.* Water to hydrogen and oxygen (Fig. 88, page 45).

#### Some general methods for collecting and storing gases.

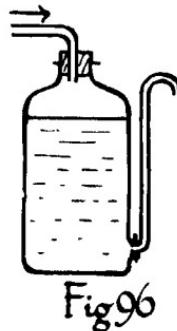
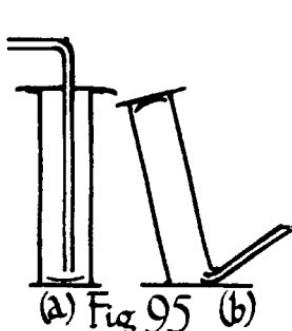
1. In gas jars or tubes over a liquid, usually water, in a pneumatic trough (Fig. 94).

2. By displacement of air (Fig. 95).

For gases heavier than air, see Fig. 95 (a).

" " lighter " " 95 (b).

3. Passing into an aspirator (Fig. 96). By this arrangement the volume collected or used can be measured exactly.



#### GENERAL PROPERTIES OF GASES

All gases expand when heated and contract when cooled, and the rates at which they do so are the same in all cases—approximately  $1/273$  of the volume at  $0^{\circ}\text{C}$ . for each degree Centigrade.

This general property is summarized and expressed mathematically in the form called *Charles' Law*.

A simple piece of apparatus for its experimental verification is shown in Fig. 97. The flask A is full of gas (say air). It is kept immersed in the beaker B by a lump of lead C. The mouth of the flask is closed by a cork, through which passes a delivery tube D. When the water is heated the gas expands, the excess being driven out through the tube. The temperature is taken before heating the gas, and the heating continued until the water boils.

The expansion is measured by allowing the flask to cool with the end of the tube under water, when the water rushes back to fill the place of the expelled gas. The volume of A is measured, and then the

increase calculated for 1 c.c. for 1 degree Centigrade rise in temperature.

Accurate determinations of the coefficient of increase of volume of a gas at constant pressure can be made by using the form of apparatus illustrated in Fig. 98, which was designed by the author and used in his physics laboratory at the Mundella School, Nottingham, some twenty-five years ago.

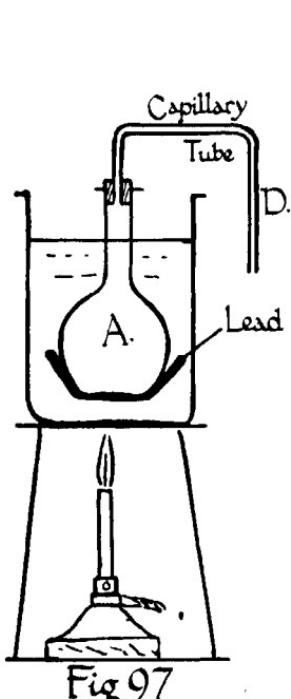
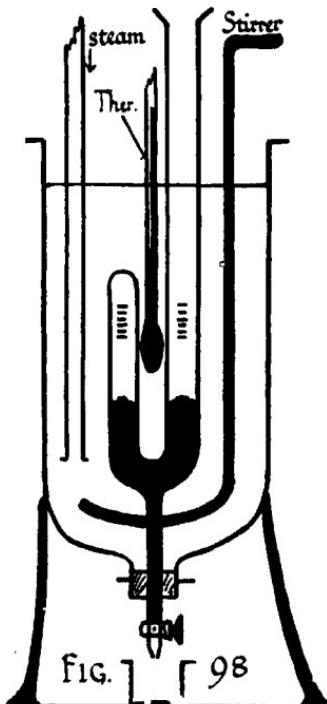


Fig. 97



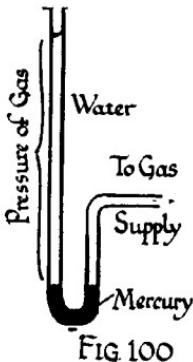
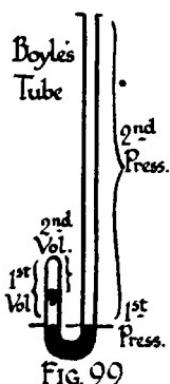
The "Constant-pressure Air Thermometer" shown in this diagram was first published by the author (and awarded first prize) in connexion with a competition organised by Newnes in their publication *Technics* in 1904. It is republished with their permission.

**Method of Using.** The gas is enclosed in one limb of a U tube by means of mercury, which can be kept at constant level by withdrawing from the tube joined to the bottom of it, or by filling in at the top of the longer limb.

The portion of the tube containing the gas is graduated so that volumes may be read, and these graduations are continued on the other limb to enable a constant level to be obtained accurately.

The bath is filled with cold water, the pressure adjusted, and the volume and temperature read. Steam is then passed in and the temperature gradually raised. At intervals of (say)  $10^{\circ}\text{C}$ . the pressure is again equalized and the volumes determined. The expansion per unit volume for unit rise in temperature may then be calculated.

**Boyle's Law.** The volume occupied by a gas is also dependent upon the *pressure* on the gas; this is illustrated in a pop-gun or air-gun. Boyle investigated the problem of "the spring of air" long ago and found that for a given mass of gas at a constant temperature the *volume was inversely proportional to the pressure*, i.e.



If the pressure was doubled the volume was halved.

" " " trebled " " made one-third.

" " " quadrupled " " one-fourth, etc.

This apparatus is illustrated in Fig. 99. Pressure gauges may be constructed on this principle. If the tube is open at the end, it is called a manometer (Fig. 100), and it can be used to measure the pressure of a gas, say the domestic gas supply, in inches of water.

**Diffusion.** The particles of which gases are composed are always in motion. They are thus able to:—

1. Mix with each other irrespective of relative densities, e.g. if a jar of hydrogen be inverted over a jar of oxygen, at the end of a few minutes each jar will be found to contain an explosive mixture of oxygen and hydrogen (Fig. 101).

2. Pass through a solid partition which contains pores, such as unglazed porcelain (Fig. 102). This phenomenon is known as diffusion.

Graham, who investigated the problem, found that the *rate of diffusion* was inversely proportional to the square root of the densities of the gases, e.g. density of hydrogen = 1, density of oxygen = 16. Square roots of these numbers = 1 and 4. Then *rates* of diffusion are 1 : ½.

#### IDENTIFICATION OF GASES

Many gases, if they are pure, can be recognized easily by certain simple tests, thus :—

- (a) Action on a glowing splint.
- (b) " " a lighted taper.
- (c) " " wet litmus paper—red and blue.
- (d) " " lead acetate paper.
- (e) " " potassium chromate paper.
- (f) " " starch paper.
- (g) " " starch iodide paper.
- (h) " " a drop of ammonia on a glass rod.

#### Gases recognized—

1. From their *appearance*—chlorine (green), hydrochloric acid (white fumes), nitrogen peroxide and bromine (brown), iodine (violet).
2. By their *odour*—chlorine (irritating), ammonia (pungent), sulphuretted hydrogen (fœtid), sulphur dioxide (suffocating).
3. By *glowing splint* test—oxygen and nitrous oxide (re-ignite).
4. By *combustibility*—hydrogen, sulphuretted hydrogen, carbon monoxide.

#### PRACTICAL EXERCISES

Try to identify the gas evolved in each of the following experiments :—

1. Heat a mixture of potassium chlorate and manganese dioxide.
2. Warm some ammonium chloride with caustic soda.
3. Act on sodium sulphite with dilute hydrochloric acid.
4. Heat lead peroxide with strong hydrochloric acid.
5. Warm some aluminium with caustic soda solution.
6. Heat oxalic acid crystals with strong sulphuric acid.

#### II. THE ATMOSPHERE.

Air is the most important of man's necessities. We can live several days without food, several hours without water, but not more than two or three minutes without air. Its physical and chemical properties have been subjects of investigations for generations, and the following conclusions have been established :—

(a) It is necessary to life and combustion. All other gases are useless, poisonous, or asphyxiating so far as animals are concerned. To get a fire up, we must allow air to have access to the coal. To put it out, we cover it up to prevent air getting to it.

(b) Fresh air is also necessary, or

(c) Its power of supporting combustion is limited. To illustrate this, burn a candle under a bell jar (Fig. 103). Other facts illustrating the same truth are the Black Hole of Calcutta episode, the necessity for ventilation of inhabited buildings, open-air treatment of consumption.



FIG. 103

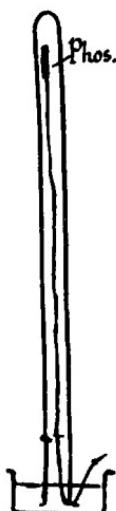


FIG. 105

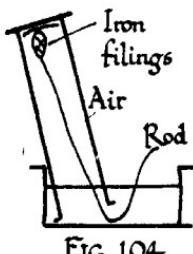


FIG. 104

(d) Boyle noticed that when metals were exposed to, or heated in contact with, air, they were altered in appearance and gained in weight, forming what he called a *calx*. Examine the calces of lead, copper, iron, zinc, mercury.

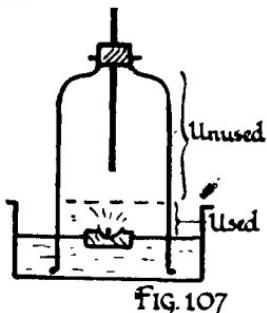
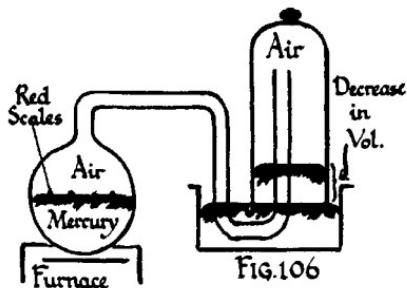
(e) In these processes a *portion* of the air is used up. This can be demonstrated by putting a muslin bag of iron filings in a jar of air and inverting over water for several days (Fig. 104), or using phosphorus in a longer tube (Fig. 105). Lavoisier's original apparatus is shown in Fig. 106. He treated mercury for several days in his retort, and found that the air gradually decreased in volume until there was no further contraction, and that specks of red appeared on his mercury. When these specks were strongly heated, they yielded a gas which gave the same volume as that lost by the air, but this gas was much more active.

than original air, and was identical with a gas, named oxygen by him, obtained by other methods.

(f) The portion left does not act like original air, and was called dephlogisticated air, or azote, now named nitrogen. Therefore

(g) The atmosphere is considered to consist of two gases at least.

To determine the proportion in which these two gases exist. (The original method of Lavoisier is unsuitable for general use.) The simplest way is to burn some phosphorus in a bell jar of air standing in some water. The phosphorus can be ignited by warming the end of



a metal rod and quickly inserting the stopper which carries it. The volumes of original and residual air should be marked with strips of gummed paper (Fig. 107).

*Accurate method.* Use the apparatus shown in Fig. 108, which consists of a bottle containing a known volume of air, fitted with a rubber stopper through which pass the delivery end of a stoppered burette and a manometer tube.

Put into the burette a little strong solution of "Pyro" in water, and fill up with caustic soda solution. Read level of liquid in the burette. Allow a little pyro-soda to pass into the bottle and shake carefully. Some of the oxygen is absorbed, and, the pressure of air in

the bottle being thereby decreased, one arm of the mercury falls, and the other rises in the manometer. Allow more solution to flow into the bottle until the surfaces of the mercury columns become permanently level. Read the burette, and so find the volume of liquid passed into the bottle—this is equal to the volume of oxygen in the original volume of air. Calculate the percentage. The volume of nitrogen is found by difference. In round numbers the result should be 21 per cent. of oxygen, 79 per cent. nitrogen by volume.

*Other constituents.* Careful and exact experiments have shown that, besides oxygen and nitrogen, there are present in air (a) water vapour; (b) carbon dioxide; (c) argon; (d) traces of nitric acid, ammonia, ozone, etc.; (e) solid matter; (f) living organisms known as bacteria or germs.

The presence of *water vapour* is proved by rain, clouds, etc., and by the exposure of hygroscopic substances to air, e.g. strong sulphuric acid increases in weight and gets weaker.

Solid caustic soda or potash becomes wet.

Dried cobalt chloride (on filter paper) becomes pink in a damp atmosphere.

At 0° C. 1 cu. metre of air can hold about 5 grams of water vapour.

“ 10° C. 1	”	”	”	”	9·5	”	”	”
“ 16° C. 1	”	”	”	”	14·0	”	(1·4%)	”
“ 20° C. 1	”	”	”	”	17·0	”	”	”
“ 30° C. 1	”	”	”	”	20·0	”	”	”

Therefore its point of saturation depends upon the temperature. Air near its point of saturation is said to be humid, and it has a very enervating effect on the human system, particularly at the higher temperatures.

A certain degree of humidity is *necessary* in a weaving shed. For certain classes of goods the ordinary atmospheric condition of Lancashire is sufficient to ensure it, but for others it is necessary to supply an artificial humidity. To guard against this being carried to excess, the manufacturer who steams is compelled to determine and record three times per day data from which the percentage humidity of his shed can be calculated.

*To determine relative humidity.* The only accurate way for finding humidity, is to pass a known volume of air through a desiccating agent which can be weighed before and after the experiment. This necessitates accurate apparatus, and requires considerable time, during which the proportion of moisture present can have changed considerably. Therefore it is not usual to use this method, but one due to Mason known as the wet and dry bulb hygrometer, in conjunction with Glaisher's Tables, which have been compiled from results obtained by an accurate method.

Two thermometers are needed, the bulb of one being covered with muslin, which is kept wet by immersion in a small vessel of water. If the air be not saturated with moisture, evaporation takes place from this muslin, with the result that the temperature of this thermometer is lowered. The drier the atmosphere, the more the evaporation and consequently the lower the reading will be (Fig. 109).

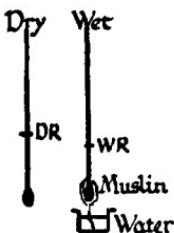


FIG. 100

To find percentage humidity.

Take the reading of each thermometer simultaneously.

Say dry bulb reading is 22° C.

" wet " " " "  $18^{\circ}\text{C}$ .

Find the difference  $22 - 18 = 4^{\circ} \text{ C.}$

Use the table as follows :—

Go to the line for the dry bulb reading, i.e. 22.

and in this line find two factors :—

(a) That given in the 0 column = 19.7.

(b) " " " " difference 4 column =

(b) " " " difference + column = 12.9.

Then percentage humidity =  $\frac{b}{a} \times 100$  or  $\frac{12.9 \times 100}{19.7} = 65.5$  per cent.

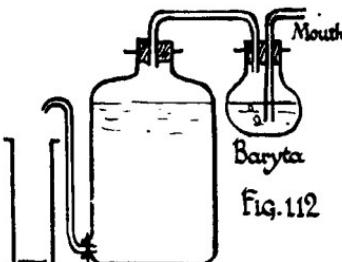
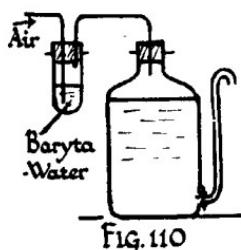
## **HUMIDITY TABLE**

Reading of Dry Bulb in deg. C.	"Factors" when Differences between wet and dry Bulb is							
	0	1	2	3	4	5	6	7
10	9.2	8.0	6.8	5.7	4.6	3.5	2.5	1.5
11	9.8	8.6	7.4	6.2	5.1	4.0	2.9	1.9
12	10.5	9.2	8.0	6.8	5.6	4.5	3.4	2.3
13	11.2	9.8	8.6	7.3	6.2	5.0	3.9	2.8
14	11.9	10.6	9.2	8.0	6.7	5.6	4.4	3.3
15	12.7	11.3	9.9	8.6	7.4	6.1	5.0	3.8
16	13.5	12.1	10.7	9.3	8.0	6.8	5.5	4.3
17	14.4	13.0	11.5	10.1	8.7	7.4	6.2	4.9
18	15.4	13.8	12.3	10.9	9.5	8.1	6.8	5.5
19	16.4	14.7	13.2	11.7	10.3	8.9	7.5	6.2
20	17.4	15.7	14.1	12.6	11.1	9.7	8.3	6.9
21	18.5	16.8	15.1	13.5	12.0	10.5	9.0	7.6
22	19.7	17.9	16.2	14.5	12.9	11.4	9.9	8.4
23	20.9	19.0	17.3	15.6	13.9	12.3	10.8	9.2
24	22.2	20.3	18.4	16.6	14.9	13.3	11.7	10.1
25	23.6	21.6	19.7	17.8	16.0	14.3	12.7	11.1
26	25.0	22.9	21.0	19.0	17.2	15.4	13.7	12.1
27	26.5	24.9	22.3	20.3	18.4	16.6	14.8	13.1
28	28.1	25.9	23.7	21.7	19.7	17.6	16.0	14.2
29	29.8	27.5	25.3	23.1	21.1	19.1	17.2	15.3
30	31.6	29.2	26.9	24.6	22.6	20.5	18.5	16.6

Home Office Regulations provide that no artificial humidification will be allowed in any weaving shed where steaming is carried on when the wet bulb reading of the hygrometer exceeds 75 deg. Fah. (i.e. 24° C.). Previously steam was allowed to be infused until the wet bulb thermometer registered as high as 91° F. or 33° C. The readings have to be entered three times a day jointly by representatives of the firm and the operatives.

The presence of carbon dioxide is proved by aspirating a good volume of air through baryta water or lime water (Fig. 110), which is turned milky by the gas. It is derived from the combustion of carbon and carbon compounds, and respiration of animals. The assimilation of it by plants keeps the gas from accumulating in excess of .04 per cent. by volume. It may however be locally in excess, e.g. in crowded rooms, smoky towns, mines, etc.

Carbon dioxide in air can be estimated approximately by the Angus Smith test, and accurately by the Pettenkofer method. The other



constituents require more accurate and refined methods for their identification.

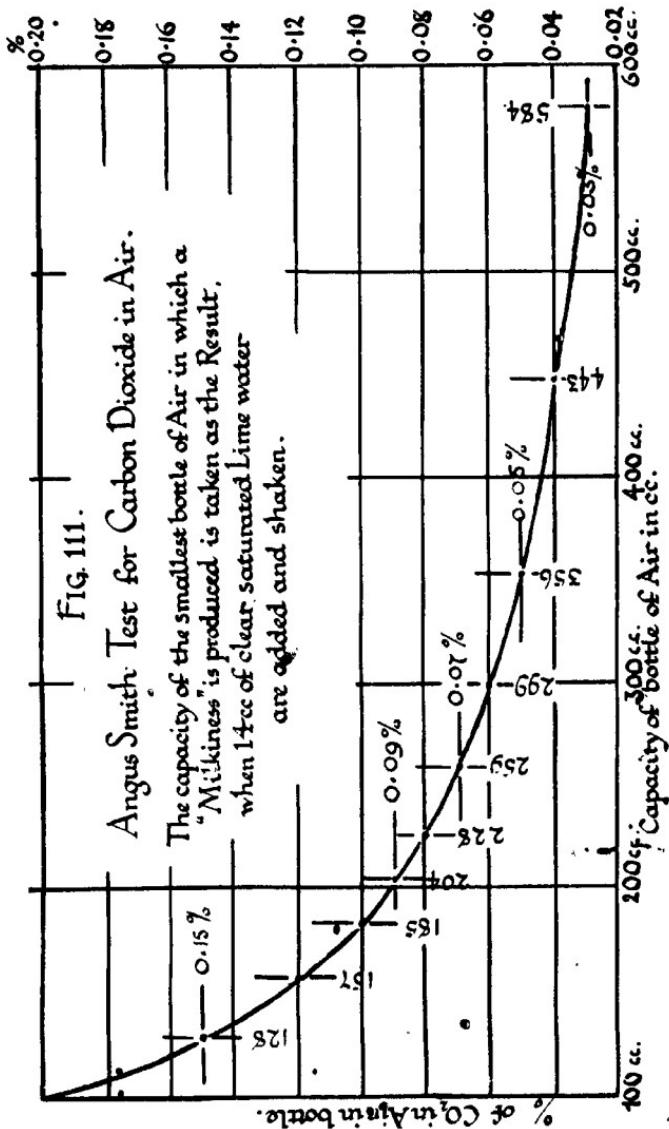
**Estimation of carbon dioxide in air by Angus Smith method :** Take bottles of various capacities ranging from 100 c.c. to 580 c.c. and fill them with samples of the air to be tested. To each add 14 c.c. of a clear saturated solution of lime water and shake. Note the *smallest* bottle in which a milkiness is produced. Refer to the graph (Fig. 111) and find the percentage of carbon dioxide which is given for its capacity, e.g. if 185 c.c. then percentage = .1 ; 300 c.c. = .06 per cent. ; 445 c.c. = .04 per cent.

At its best, Smith's test is but a rough-and-ready method.

**The Pettenkofer Method** in one or other of its numerous modifications is always used if we require a quantitative result. Its manipulation is delicate and requires much practice before accuracy is attained, particularly in normal air determinations.

As an exercise for the beginner, good and interesting results can be obtained with it for *expired air* working as follows :—

Arrange apparatus as shown in Fig. 112. In the small flask put 20 c.c. of semi-saturated baryta water, made by shaking baryta with



distilled water, and when saturated decanting, and diluting with an equal volume of water.

Blow air from the lungs *slowly* through this solution and measure the water expelled from the aspirator, which will be approximately equal to the volume of expired breath. A suitable amount to pass through is 1,000 c.c. When finished, disconnect the flask.

In a similar flask put another 20 c.c. of the original baryta water, and to each add 2 or 3 drops of an indicator called phenolphthalein. A pink colour is produced. Now from a burette run in a standard solution of oxalic acid containing 5·65 grams of the pure acid dissolved in one litre of water.

Find the volume required *just* to destroy the pink colour in each case. It will be found that less is required by the baryta through which expired air has passed. The difference can be utilized to measure the amount of carbon dioxide which has passed through.

Suppose the difference = 37 c.c.

Now 1 c.c. of oxalic acid of above strength = 1 c.c. carbon dioxide.

Then 37 c.c.   ,   ,   ,   ,   = 37 c.c.   ,   ,

which is present in (say) 1,000 c.c. of expired air, i.e. = 3·7 per cent.

The amount of carbon dioxide in air can be taken as an index to the efficiency of the ventilation of a building. A high percentage of the gas means a vitiated and unhealthy atmosphere.

A Parliamentary Committee Report issued in 1909 said, "The greatest evil in a mill is the lack of efficient ventilation," and this remark could well be extended to English buildings generally, including dwelling-houses, and particularly bedrooms.

Principles of ventilation are well understood by scientific experts, but the application of them seems to make very little headway, due to general ignorance or carelessness and stupidity on the part of the British public.

One of the purest atmospheres in a public building in the United Kingdom is that of the British House of Commons. This is due partly to the fact that (as a rule) the number of people in it is small, but chiefly because it is ventilated on a system.

The "draught" is supplied by a shaft running up one of the famous towers, and the air drawn in is washed, warmed, filtered, and then allowed to enter under the benches. It rises towards the ceiling and is extracted at such a rate that no strong current is produced.

Air which contains *dust* is distinctly dangerous to health. Professor Tyndall once made the famous remark, "Shut your mouth, and save your life," meaning thereby that you should breathe through the nose, the hairs in which arrest many of the solid particles.

Every solid particle is a small world on which may rest hundreds

of spores, microbes, or germs capable of producing pulmonary and gastric troubles and, it may be, disease and death.

A simple piece of apparatus which can be used to trap and estimate the bacteria in air is shown in Fig. 113.

A is a small tube containing sterile water through which air may be drawn by attaching to B, which in its turn is connected to C by means of a flexible rubber tube.

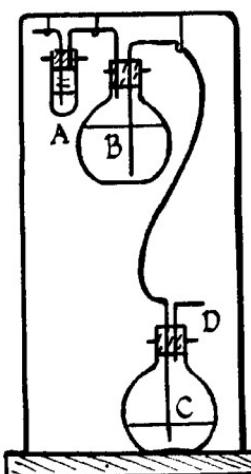


FIG 113

By suction at the tube D the water can be started running from B to C, which thus causes a similar volume of air to bubble through A.

By interchanging the flasks when C is empty, and repeating the process, more air is bubbled through the water in A, and if the volume of water used is known the exact volume of air passed through can be calculated.

An aliquot part of the water in A is plated on sterilized gelatine or agar and cultivated, when each bacteria will produce a "colony" if incubated at a suitable temperature.

## SECTION VII

### I. OXYGEN

OXYGEN is the most abundant element in the world—half the crust of the earth, eight-ninths of water, and one-fifth of air is composed of it.

It was originally obtained from calx of mercury, first as an "unidentified spirit" by Eck de Sulzbach in 1489, and later by Priestley in 1774. Scheele had prepared the gas in 1773, but had not published the fact. The gas can be prepared in a great many ways, the most important of which are :—

1. By heating oxides which evolve the gas, e.g. mercuric oxide,

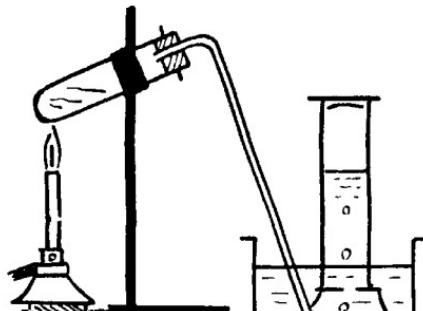


FIG. 114

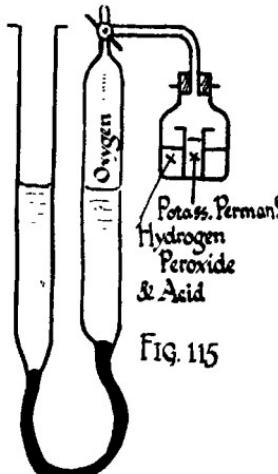


FIG. 115

manganese dioxide, silver oxide, barium peroxide, red lead, lead peroxide, etc.

2. By heating potassium chlorate, particularly if mixed with manganese dioxide (Fig. 114). This is the usual laboratory method of obtaining the gas.

3. By heating certain substances with sulphuric acid, such as potassium dichromate, potassium permanganate, manganese dioxide.

4. From bleaching powder by warming it with water to which has been added a little cobalt nitrate (to produce cobalt oxide, which acts as the decomposing agent).

5. By evaporation of liquid air. Nitrogen boils at a higher temperature than oxygen, so, if liquid air be distilled under certain conditions, oxygen is first evolved. This is how oxygen is now prepared commercially.

6. By the interaction of solutions of potassium permanganate and hydrogen peroxide in the presence of sulphuric acid (Fig. 115). Inside the small bottle is a smaller one. Hydrogen peroxide is put in one, acidified potassium permanganate in the other, and the bottle is then connected with the Hempel burette. On tilting the apparatus, the liquids mix, oxygen is evolved and collects in the limb of the burette.

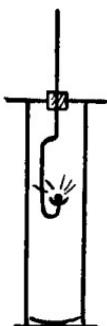


FIG. 116

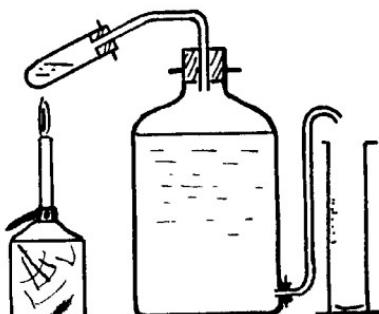


FIG. 117

Oxygen is a very active gas; glowing splints, carbon, phosphorus, sulphur, and even iron wire burn in it with great energy, in most cases to form oxides. The operation is conducted with a deflagrating spoon as shown in Fig. 116.

*To determine the volume of oxygen evolved by heating a substance which yields it, use the apparatus shown in Fig. 117. Weigh the empty tube, put the substance in and reweigh. Attach it to the aspirator as shown and collect the water expelled from the delivery tube. Measure it and calculate the volume obtained from 1 gram of the substance.*

As an exercise, use potassium chlorate.

*To find the weight of oxygen expelled when a substance is heated, or to find the weight which combines with a substance when heated in air, use a crucible, etc. (Fig. 44, page 12).*

## EXERCISES

- Find the weight of oxygen expelled from 100 grams of potassium chlorate when strongly heated.
- Take a piece of magnesium ribbon about 2 feet long, clean it with sand-paper, weigh it and divide into two equal parts. Burn one piece in air by heating on a crucible lid over a bunsen flame. Carefully collect all the calx and weigh.

Compare with the weight of magnesium used.

Dissolve the other piece in two or three drops of dilute nitric acid on a crucible lid. Heat to dryness and until all brown fumes are driven off. Again collect the white powder, weigh and compare with the weight of calx from the previous experiment. Taste to see if it is magnesia (magnesium oxide).

## II. OXIDATION AND OXIDES

The act of chemical union with oxygen is known as oxidation. If the substance which is oxidized is an element, the compound produced is called an *oxide*. An older name is calx.

Nearly every element combines with oxygen under one condition or another, directly or indirectly, to form an oxide, and some produce 2, 3, 4, or even 5. More than 100 different oxides have been prepared.

*Iron* oxidizes in moist air to form iron oxide; it is termed rusting.

*Lead and copper* both tarnish in air, especially if heated, due to oxidation.

N.B.—Tarnishing does not always denote oxidation, e.g. the tarnish on silver is due to the formation of a sulphide, not an oxide.

*Phosphorus, sodium, potassium* need only to be exposed to air for a few seconds to bring about oxidation.

*Aluminium, zinc, magnesium, tin, mercury* do not easily oxidize to a notable degree in ordinary air at ordinary temperatures.

Other oxides may be produced by *calcination* or roasting, i.e. heating in air or oxygen. *Mercuric oxide, magnesium oxide, tin oxide, zinc oxide*, the oxides of carbon and sulphur may all be prepared in this way.

Another method which may be employed is to use substances which will readily yield oxygen, such as nitric acid, bleaching powder, potassium chlorate, hydrogen peroxide, etc., and which are therefore called *oxidizing agents*, e.g.:-

- Add strong nitric acid to tin, white tin oxide is formed.
- Take some cobalt nitrate solution in a test tube. Add two or three drops of sodium hypochlorite (note the formation of black oxide of cobalt). Add it to an emulsion of bleaching powder and water and gently warm. Test the gas evolved. What is it?
- Heat, with constant stirring, a small piece of lead in a crucible.

lid. Note the changes that occur. Try to get some litharge. Did you ? If so, state what it was like.

4. Oxidize some red lead by warming it with dilute nitric acid. Describe the product. How many oxides of lead have you seen ? Compare and contrast them.

5. Take a few drops of mercuric chloride. (Be careful : this is a deadly poison.) Add caustic soda solution. What happens ? Filter off the precipitate. Carefully dry it and then heat it strongly in a small ignition tube. Can you identify the gas evolved ? If so, state what it is.

### III. COMBUSTION

is often an act of oxidation. Lavoisier was the first chemist to explain it as such. The old theory was that when a substance burned it *lost* something, known as phlogiston, and it was then unable to burn again until it had been treated with a substance rich [in phlogiston, such as carbon, which supplied it with some.



Lavoisier's theory of combustion was, that when a substance burned it *gained* something, that something being oxygen, obtained either from the air or a substance capable of yielding it.

This explains why substances burn in ordinary air or oxygen, and why they refuse to burn in the inactive four-fifths of air which contains no oxygen.

Apparently when a candle burns it loses weight, but if an apparatus be arranged for it to burn in, so that all the *products of combustion* are trapped and also weighed, it is found that there is a gain and not a loss (Fig. 118).

Sulphur and carbon burn in gunpowder at the expense of oxygen in nitre, which is one of the constituents of gunpowder.

As a rule, chemical combination with oxygen results in the production of a large amount of heat, sometimes sufficient to make the body red, or even white hot, and sometimes flame is produced.

This is very liable to occur in heaps of oily cotton waste. The oil is oxidized by the oxygen of the air and gradually the temperature is raised until it is sufficient to produce and ignite a vapour from the oil ; and so we get the starting of a fire, which still is far too common in Lancashire mills.

### IV. BREATHING

is also an act closely connected with oxidation, being a process for supplying oxygen to the blood and removing carbon dioxide from it.

The warmth and sustenance for the human body is produced by the slow oxidation in the tissues of the carbon and hydrogen taken as food.

This results in the formation of a large amount of carbon dioxide and water—the former being carried by the blood to the lungs, where it is liberated into the air chambers and then expelled through the mouth in the act of breathing.

For this purpose the average volume of air needed by an adult is 1,500 gallons per day.

## V. OXIDES

of the different elements differ in various ways, e.g. state, effect produced by heat, solubility in liquids.

**Solids.** Lime (calcium oxide), baryta (barium oxide), zinc oxide, oxides of iron, magnesium, copper, tin, mercury, lead.

**Liquids.** Water (oxide of hydrogen), nitrogen peroxide.

**Gases.** Carbon monoxide, carbon dioxide, sulphur dioxide, nitrous oxide, nitric oxide, ozone (oxide of oxygen).

When heated, some evolve oxygen, e.g. mercuric oxide, red lead, silver oxide, chromium trioxide, and all peroxides.

Some do not yield oxygen, e.g. lime, baryta, alumina, sand, zinc oxide, water (except at very high temperatures).

They vary very much in respect to solubility in water and other common solvents :—

*Very soluble in water.* Phosphorus pentoxide, hydrogen peroxide, sulphur trioxide, sodium oxide, potassium oxide.

*Moderately soluble in water.* Carbon dioxide, sulphur dioxide, baryta.

*Slightly soluble in water.* Lime, iron oxide.

*Nearly insoluble in water.* Lead oxide, sand.

*Soluble in dilute nitric acid.* Bismuth, copper, zinc oxides, barium peroxide, litharge.

*Insoluble in dilute nitric acid.* Tin oxide, antimony trioxide, lead peroxide, sand.

Oxides are usually classified as :—

1. **Acidic Oxides.** Those which combine with the elements of water to form acids—e.g. oxides of sulphur, carbon dioxide, oxides of nitrogen (usually non-metals).

2. **Basic Oxides.** Those which combine with the elements of water to form bases (metallic oxides)—e.g. sodium and potassium oxides, lime, baryta.

3. **Peroxides**—which yield oxygen on heating and which do not react with water to form acids or bases, e.g. manganese dioxide, lead peroxide, barium peroxide, hydrogen peroxide.

## SECTION VIII

### I. ACIDS, ALKALIS, BASES, SALTS

**T**Hese are important classes of compounds which are very closely related one to the other.

Some hundreds of acids are known, nearly a score of alkalis, about 100 bases, and thousands of salts. Each class exhibits certain distinctive characteristics by means of which it may be identified.

**Acids** possess a sour taste; change the colour of certain natural colours; react with alkalis and bases to produce salts; always contain the element hydrogen and conduct electricity in solution.

It was stated by Lavoisier that oxygen was the real acidifying principle, but this has been disproved.

**Alkalis** have a caustic taste and a soapy feel; they also change certain colours, react with acids to form salts, and as a rule are very soluble in water.

**Bases** are compounds which neutralize acids to form salts and contain a metallic element.

**Salts** are compounds in which the hydrogen of the acid has been replaced, partly or entirely, by a metal. They are usually (a) of a solid and crystalline nature, (b) neutral to litmus, (c) when in solution decomposed by the passage of an electric current.

Salts may be *normal*, in which all the replaceable hydrogen has been replaced by a metal; *acid or hydrogen*, in which some has not been replaced; *basic*, which contain a greater quantity of the metallic radicle than is sufficient to replace completely the hydrogen; and *double*, which contain more than one metal.

Copper sulphate, zinc chloride, magnesium chloride, common salt, Glauber salt, Epsom salt, sodium carbonate, are examples of normal salts; sodium bicarbonate, nitre cake are acid salts; copper carbonate, white lead are basic; and alum is a double salt.

Examine samples of the following:—

(a) *Acids*. Acetic, boric, citric, hydrochloric, nitric, oleic, oxalic, palmitic, salicylic, stearic, sulphuric, tannic, tartaric.

(b) *Alkalies*. Caustic soda, caustic potash, quicklime, ammonia, washing soda, baryta.

(c) *Bases.* Metallic oxides such as black copper oxide, litharge, zinc oxide, mercuric oxide.

To study the action of acids and alkalis on coloured bodies, use solutions of the following *indicators* :—

- (a) Litmus in water.
- (b) Methyl orange in water.
- (c) Phenolphthalein in 50 per cent. alcohol.
- (d) Lacmoid in 50 per cent. alcohol.
- (e) Congo red on paper.
- (f) Cochineal in 25 per cent. alcohol.

Note that *acids* may turn blue litmus to red ; lacmoid to red ; methyl orange to pink ; cochineal yellowish red ; Congo red to blue. Further, certain of these indicators are not affected by certain *acids*, e.g. the fatty acids—oleic, stearic, and palmitic—do not change methyl orange.

Note that *alkalis* turn litmus and lacmoid to blue ; methyl orange to yellow ; phenolphthalein to pink ; cochineal to violet.

Note that *bases* (other than alkalis), as a rule, have no action on indicators.

**Preparation of Salts.** When acids and alkalis or bases react, a chemical change results, a salt being formed. This is termed *neutralization*, e.g. :—

1. Take some dilute hydrochloric acid in a dish and add a drop or two of litmus. Now add caustic soda, drop by drop, till the litmus is just turned purple. Evaporate down to dryness. The residue is common salt.

2. In a similar way neutralize caustic potash with nitric acid and obtain solid nitre.

Salts may be obtained also by the following methods :—

3. Dissolve copper oxide in hot dilute sulphuric acid, evaporate to a small bulk, and cool to crystallize out the copper sulphate.

4. Dissolve zinc in dilute hydrochloric acid, and concentrate to obtain zinc chloride. Note that so long as there is any free acid in this solution, Congo red is turned blue, but when entirely neutralized no effect is produced on the indicator.

Salts of hydrochloric acid are called chlorides.

„	nitric	„	„	nitrates.
„	sulphuric	„	„	sulphates.
„	acetic	„	„	acetates.
„	citric	„	„	citrates.
„	carbonic	„	„	carbonates.
„	tartaric	„	„	tartrates.
„	oxalic	„	„	oxalates.

Salts of palmitic acid are called palmitates.

“ oleic                ”         oleates.

“ phosphoric        ”         phosphates.

Many salts are without action upon indicators, but some do affect them, e.g. :—

Copper sulphate turns blue litmus red, so does zinc chloride, while sodium carbonate turns red litmus blue.

Fats may be looked upon as salts (esters they are called) of certain organic acids, of which an enormous number are known. Here the base is glycerine, not a metallic oxide, although metallic oxides will combine with the fatty acids.

Palm oil is largely a glyceride of palmitic acid.

Mutton fat contains a large quantity of glycerine stearate.

Lard contains a considerable amount of glycerine oleate ; while

Beef fat is a mixture of the glycerides of stearic, oleic, and palmitic acids (chiefly the former).

Lead plaster is made by boiling olive oil with litharge, and is really lead oleate.

Compounds of metallic oxides and fatty acids are sometimes termed metallic soaps, and their production is often a cause of serious trouble during sizing operations if incompatible ingredients have been used in the mixing.

Esters are all *hydrolyzed* on boiling with caustic alkalis, i.e. the glycerine is regenerated and the alkali salt of the fatty acid formed. This new salt is called a *soap*, the process often being termed on that account *saponification*.

Soft soaps are usually potash soaps, and hard soaps are generally soda soaps, but not always so, as the nature of the fat or oil used has a considerable influence on the physical properties of the soap formed.

## II. SULPHURIC ACID

It is almost impossible to overrate the great commercial and industrial importance of this well-known acid. South-west Lancashire and North-east Wales are full of factories for its manufacture, and the normal annual production of the acid in Great Britain alone is two million tons out of four million tons consumed yearly.

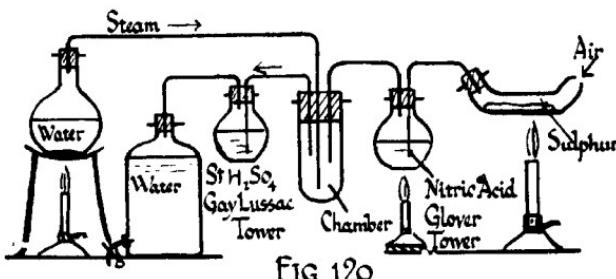
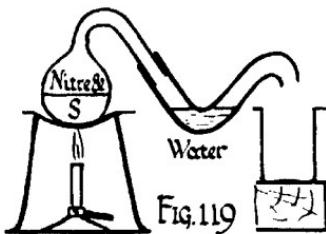
It was prepared originally from ferrous sulphate (green vitriol) by the action of heat, which expelled sulphur trioxide and water vapour, that united in the receiver to form the acid and which, being of an oily nature, was named *oil of vitriol*.

In the seventeenth century it was prepared by heating sulphur and nitre, which can be illustrated by using the apparatus shown in Fig. 119. The mixture of sulphur and nitre is gently heated until the

sulphur burns in the nitre. Sulphur trioxide distils over and is collected in the water contained in the bend, thus forming a dilute solution of sulphuric acid.

The first sulphuric acid works was established in Richmond in 1740. After that time the industry rapidly extended and the method of manufacture was greatly improved. Gay Lussac devised a means of trapping the most valuable by-product and Glover invented a method for using it over again.

The cost of manufacture was thus greatly reduced, and the use of the acid became much more general. Further reduction in the cost of production was effected (but at the expense of purity) by using pyrites instead of sulphur to yield the sulphur dioxide.



The principle of the English method of manufacture is illustrated in Fig. 120.

Sulphur is heated in a hard glass tube through which air is passing. This forms sulphur dioxide, which is carried forward with excess of air through a flask containing nitric acid, which when warmed liberates nitric oxide. The mixture of sulphur dioxide, air and nitric oxide now passes to the large boiling-tube, where it meets with steam, with the result that sulphuric acid is produced and falls to the bottom of the tube.

The nitric oxide acts as the agent which brings about the change, and is not itself used up. If the gases from the tube are passed through

a flask containing strong sulphuric acid, the nitric oxide will be absorbed and not lost to the process. It can be used over again by putting the liquid into the flask which contains the nitric acid.

These flasks correspond to the Glover and Gay Lussac towers in the manufacturing plant, details of which can be obtained from the larger chemistry textbooks.

The current through the apparatus is maintained by running water from the aspirator.

**The Contact Process** for the manufacture of sulphuric acid: The purest and strongest acid is now made by a catalytic contact process, on the perfecting of which enormous sums of money and much labour and research have been expended.

The laboratory method of working is simple to carry out and easy to understand (Fig. 121), but on the manufacturing scale it is much more difficult to accomplish successfully.

A catalyst is a substance which acts as an accelerator of a chemical change. The precise action is not definitely known. Ostwald compares it to a "chemical oil." Manganese dioxide and cobalt oxide both act as catalysts in the preparation of oxygen from potassium chlorate and bleaching powder respectively.

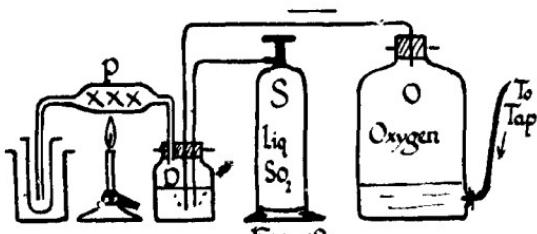


FIG. 121

The contact process has been worked principally on the Continent for the production of an acid which was necessary to the dye industry, and which could not be produced by the English chamber process.\* There is no doubt but that it will ultimately displace the older method, particularly as during the last few years other catalytic agents besides platinum have been used successfully.

For the experimental illustration oxygen gas from an aspirator O and sulphur dioxide from a siphon S are forced through strong sulphuric acid in a bottle D, to dry them. They are then passed over gently heated platinized asbestos in a hard glass tube P. Here they combine to form sulphur trioxide, which is led into water W, thereby forming sulphuric acid.

Sulphuric acid comes into the market in various strengths, usually packed in carboys (Fig. 79, page 30).

Chamber acid has a strength of 62 per cent. to 70 per cent. and is used for making salt cake and fertilizers. Its sp. gr. is about 1.6. An acid of 78 per cent. to 80 per cent. is used for many technical purposes, including the manufacture of superphosphate. Its sp. gr. is about 1.72. This is collected at the foot of the Glover tower.

Double oil of vitriol (D.O.V.) has a sp. gr. up to 1.84 and a strength between 93.5 per cent. and 98.3 per cent. The impure acid is often termed brown oil of vitriol (B.O.V.), due to its colour (sp. gr. 1.72).

"Commercial" acid will always contain impurities such as iron, arsenic, lead, and copper. It should never be used in the preparation of food-stuffs or drugs.

By the contact process a pure acid is produced of 100 per cent. strength, and besides this, sulphur trioxide is dissolved in it to give fuming sulphuric acid containing sometimes as much as 45 per cent. excess of the trioxide. This is used chiefly for the production of intermediate products for the manufacture of dye-stuffs.

Strong sulphuric acid, when mixed with water, produces an enormous amount of heat; on this account the acid should always be added to water, and not water to acid. Its great affinity for water can be shown in various ways, e.g. Expose a weighed dish of it to air and weigh again after several hours; it will have increased considerably, and have become weaker. Add some to a little starch or sugar; it is dehydrated, leaving a black mass of carbon.

The test for the identification of sulphuric acid is to add a few drops of nitric acid and then a solution of barium nitrate. If a white precipitate is produced, the acid or one of its salts is present.

The use of barium chloride is not suitable in the presence of lead, which sulphuric acid is always liable to contain.

*Uses.*—Among the enormous number of uses to which sulphuric acid is put, some of the most important are. Manufacture of chemicals such as sodium carbonate, hydrochloric acid, nitric acid, alum, phosphorus; of explosives of all kinds; of fertilizers, such as sulphate of ammonia; of superphosphate; of artificial silk; in the dyeing, bleaching, and electroplating industries.

### III. NITRIC ACID

or aqua fortis (which means strong water) was known to the alchemists and was largely used for dissolving metals, nearly all of them being soluble in it.

It is a compound containing 1.6 per cent. hydrogen, 22.2 per cent. nitrogen, and 76.2 per cent. oxygen. Like sulphuric acid it has an enormous industrial application.

## PREPARATION

1. From nitre or saltpetre and sulphuric acid. Nitre is potassium nitrate. When it is heated with strong sulphuric acid it is decomposed, nitric acid being liberated as a gas. If this gas is cooled it condenses to liquid nitric acid. On a small or laboratory scale the operation is usually performed as shown in Fig. 122, which should require no further explanation.

2. From Chile saltpetre and sulphuric acid. Nitre is too expensive and too rare to use on the commercial scale. Therefore the cheaper

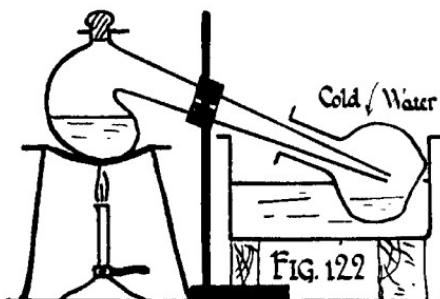


FIG. 122

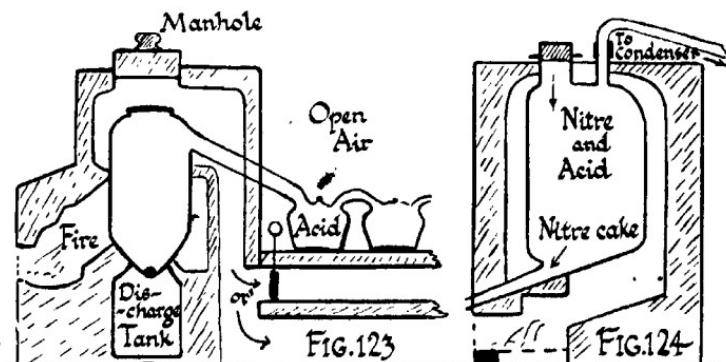


FIG. 123

FIG. 124

sodium nitrate is used in its place, and instead of glass vessels, iron retorts and porcelain bottles are employed.

Fig. 123 shows in section the usual English plant. A more modern retort, which is in use in America, is shown in Fig. 124. At the end of the condensing bottles a tower filled with coke is sometimes placed, down which trickles water. This absorbs the last trace of nitric acid, which would otherwise escape into the air.

The chemicals used are Chile nitre which has been purified until it

contains 98 per cent. to 99 per cent. of sodium nitrate and is free from sodium chloride, and sulphuric acid of sp. gr. 1·38.

These will give a nitric acid up to a sp. gr. of 1·38. For stronger acid, a stronger sample of sulphuric acid must be used.

The reaction between moderately hot sulphuric acid and sodium nitrate results in the production of nitric acid and sodium hydrogen sulphate.

If the temperature be increased further, more nitric acid would be liberated with the production of sodium sulphate (salt cake or Glauber salt), but the manufacturer finds that the high temperature at which the operation must be conducted results in the decomposition of a large quantity of the nitric acid thereby produced.

Consequently it is usual to stop at the first reaction, and the chemical which is drawn from the retort is sodium hydrogen sulphate—technically known as *nitre cake*.

Nitre cake can be used instead of sulphuric acid in certain industrial applications, "e.g. bleaching, the manufacture of baking powder, etc. (See Partington, "Industrial Chem.", pages 161, 162; or "J.S.C.I.", 35, 857, 1916; or "Chem. Tr. Journ.", 1916, 28, 109, 393, for full lists.)

3. From the air. The beds of natural nitrates in India, Chile, Egypt, etc., are being exhausted rapidly, and nearly twenty-five years ago Sir William Crookes suggested the inexhaustible air as a source of supply.

When a mixture of oxygen and nitrogen is "sparked" electrically under certain conditions it produces oxides of nitrogen which, dissolved in water, form nitric acid (Cavendish's discovery). Crookes showed that air can be burned to nitric and nitrous acids in a powerful electric arc.

There are now several successful schemes for applying these facts to the commercial production of nitric acid and calcium nitrate. It is carried out in Norway (Norwegian saltpetre) by the Birkeland and Eyde method, in which a large flame, produced by a current of high voltage, is spread over copper-surface electrodes by the action of an electromagnet, the current alternating fifty times per second.

The nitric oxide produced is rapidly cooled and then combined with oxygen to form nitrogen peroxide, which is afterwards absorbed by water to form nitric acid, or by lime to form nitrate of lime.

During the Great War, Germany rapidly developed this and other methods until she was no longer dependent upon the natural nitre beds for the production of nitric acid, but obtained it all from the air.

Commercial nitric acid can be purified by redistillation with sulphuric acid in glass vessels, and the brown colour, which is due to nitric oxide dissolved in the liquid, removed by warming the acid to 70° C. and passing a current of carbon dioxide through it (Fig. 125).

*Chemically pure acid is made by either :-*

1. Using perfectly pure materials, or
2. Treating commercial acid with barium nitrate (to precipitate sulphuric acid) and silver nitrate (to precipitate chlorine and hydrochloric acid), and then redistilling with pure sulphuric acid at a low temperature.

#### PROPERTIES

A colourless liquid of sp. gr. varying from 1.29 (46 per cent.) to 1.41 (87.5 per cent., ordinary conc.) and 1.53 (100 per cent.).

The sp. gr. is best determined with an hydrometer (see page 29), or by using a Joly balance (see pages 29, 30).

Boiling-point, 86° C.

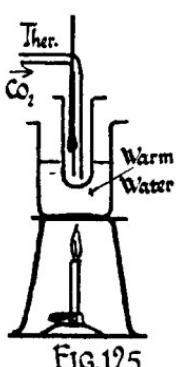


FIG. 125

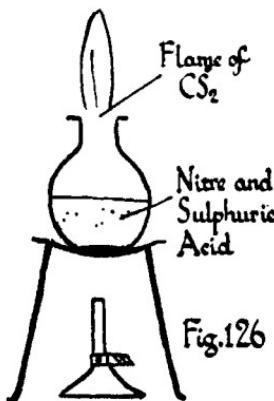


Fig. 126

Very active oxidizing agent—many interesting experiments can be performed to illustrate this property :—

1. Put some warm dry sawdust on a porcelain tray in a fume chamber, add some strong nitric acid and stir. Dense fumes of nitric oxide are evolved and the mass will in all probability burst into flame.
2. Place a small crucible on sand in the bottom of a wide beaker, and in the crucible put a few c.c. of a mixture of equal parts of strong nitric and strong sulphuric acids.
3. Put 2 grams of sugar in a large test tube, add 5 c.c. of strong nitric acid and warm. The sugar is oxidized to oxalic acid, which can be obtained by crystallization.
- Conduct this experiment in a fume chamber or out of doors, on

account of the dense fumes of the deadly oxides of nitrogen that are evolved.

4. Drop a few c.c. of the strong acid on a lump of metallic tin. It is converted into white tin oxide.

5. Take a small wide-mouth flask, put in it some nitre and conc. sulphuric acid, and gently heat it on a sand bath (Fig. 126). As soon as the flask is full of nitric acid vapour, drop in 1 c.c. of carbon disulphide, and immediately apply a light to the mouth of the flask. The carbon disulphide burns with a bright blue flame.

It is an intensely strong acid, e.g. add a drop of acid on the end of a glass rod to a litre of water, the solution will turn blue litmus red; Congo red, blue; and methyl orange, pink.

It has a very pronounced and often corrosive action on organic matter, e.g.:-

1. A feather is made yellow and destroyed if dipped in it.
2. It turns flour yellow. This can be used as a test to distinguish between pure starch and wheat or other flour. The experiment should be made in a white basin or dish.
3. It stains the fingers yellow and produces a similar effect on other bodies, e.g. indigo, proteids, silk.

In the case of silk, we can produce a permanent orange colour on it if it be soaked first in dilute nitric acid and then in ammonia solution. This is sometimes called the Xanthoproteic Reaction.

The test for the identification of nitric acid is its action on a cold strong solution of ferrous sulphate, which is turned black by it. The black substance is decomposed on heating. A nitrate will give the same reaction if it is first treated with strong sulphuric acid.

#### USES OF NITRIC ACID

1. In the preparation of gun-cotton. Make a mixture of equal volumes of strong nitric and strong sulphuric acids. Immerse in it for fifteen minutes small tufts of cotton wool. Remove with a glass rod and wash free from acid. Dry in air.

It is very inflammable; burn a small piece on a filter paper, and if it has been properly prepared it will burn away without igniting the paper.

2. In the manufacture of nitro-glycerine and dynamite, blasting gelatine, cordite, smokeless powders, etc.

3. For the production of T.N.T. (which is made by nitrating toluene), and of nitro-benzene, sometimes called oil of mirbane, used as a deodorizer in certain textile preparations and as artificial almond flavouring.

4. For the preparation of picric acid, which is made from phenol (carbolic acid) and nitric acid.

5. In the manufacture of Chardonnet artificial silk, celluloid, etc.
6. For the preparation of nitrates, such as silver nitrate (used in photography, pharmacy, for mirrors, marking ink, etc.) ; ammonium nitrate (used in the newer explosives) ; strontium nitrate (red fire) ; barium nitrate (green fire).
7. As a pickling or cleansing liquor for copper.
8. In the manufacture of dextrine from starch, and of dyes from coal-tar products.

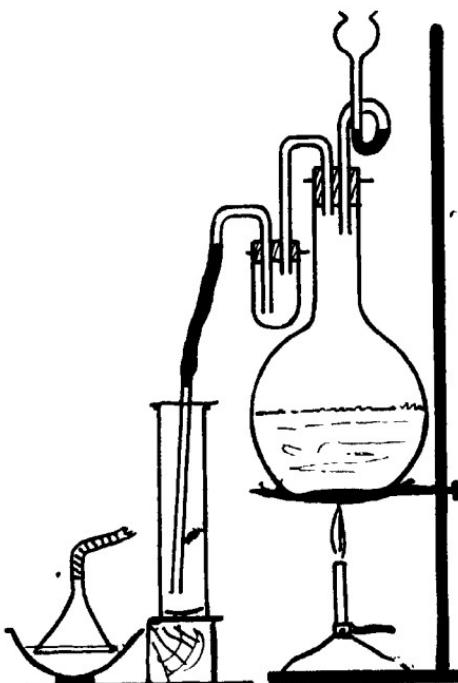


FIG. 127

#### IV. HYDROCHLORIC ACID,

also known as muriatic acid and spirits of salt, is made from common salt by heating with moderately strong sulphuric acid.

Fit up the apparatus shown in Fig. 127. In the flask put some rock salt and a little water. Add strong sulphuric acid through the thistle funnel. Hydrochloric acid gas will be evolved, at first without heat, and may be collected in the gas jar.

Fill several jars ; then attach a filter funnel to the end of the tube, and collect some of the gas in water placed in an evaporating dish. It

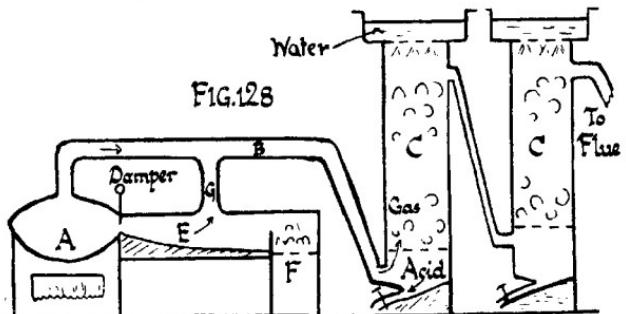
will be necessary to heat the mixture in the flask towards the end of the experiment.

With the gas perform the following experiments :—

1. Test its combustibility, and also if it will support combustion.
2. Note that it fumes in air, and much more so when in contact with a drop of strong ammonia solution held on the end of a glass rod.
3. Test its action on litmus paper and Congo red paper.
4. Test its solubility in water by inverting a jar in a pneumatic trough half full of water.

Also test with litmus paper, and with a solution of silver nitrate, the water through which the gas has been passed. A white precipitate will be produced with silver nitrate if hydrochloric acid is present.

Hydrochloric acid comes into commerce as a solution in water usually of about 32 per cent. strength (sp. gr. about 1.16), which can be diluted as required.



On the industrial scale the flask is replaced by an iron pot and the heating is done in two stages in a double furnace. Fig. 128 is a sketch of the plant used.

Salt and sulphuric acid are heated first in the pot A, and the gas evolved passes by means of the pipe B to the towers C, which are filled with coke, and down which a stream of water trickles. The solution of hydrochloric acid is drawn off at the bottom.

In the second stage the damper D is opened, the contents of A raked into the furnace E, which is heated from a coke fire at F. The gas again passes into the pipe B by means of G, and so to the collecting towers. The residue in E is called salt cake, and is chiefly sodium sulphate.

Hydrochloric acid is used in the manufacture of chlorine, and for making chlorides, and in calico-printing, bleaching, dyeing, etc.

#### EXERCISES

Prepare (a) zinc chloride by dissolving zinc in hydrochloric acid ;

(b) magnesium chloride by dissolving magnesia in the acid ; (c) calcium chloride by dissolving chalk in it.

Test the solubility of the following metals and oxides in dilute and in strong acid : Copper and copper oxide, iron and iron rust, lead and litharge, mercury and mercuric oxide.

#### V. AMMONIA

Sal-ammoniac (meaning a salt of ammonia) was first prepared by the Arabs centuries ago, by heating camel refuse in or near the temple of Jupiter Ammon, in the Libyan Desert, and so received its name from that building.

The gas itself is called ammonia or volatile alkali, and can be obtained easily from sal-ammoniac by heating it with lime or caustic soda (Fig. 129). It was formerly obtained by heating horn, and so received the name " spirits of hartshorn."

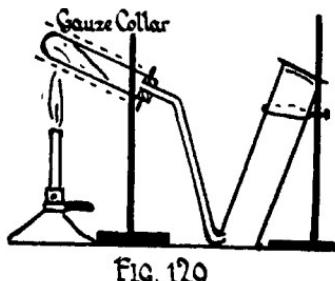


FIG. 129

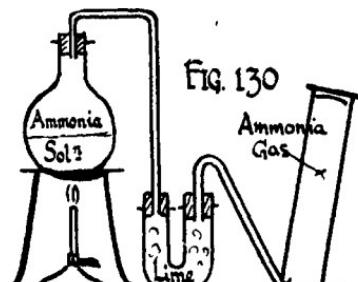


FIG. 130

At the present day an important source of our ammonia supply is gas liquor. When coal is distilled at gasworks, a large quantity of ammonia is evolved, which is collected by solution in water. This solution is known as ammoniacal, or simply gas, liquor. The liquid is mixed with quicklime and gently heated, the ammonia which is evolved being led by means of pipes into acid—either sulphuric acid to make sulphate of ammonia, or hydrochloric, to make sal-ammoniac or ammonium chloride.

An enormous amount of ammonia is now prepared synthetically from hydrogen and nitrogen. From this source it is entirely free from coal-tar products.

Ammonia is usually supplied in commerce as a strong solution in water. From this the dry gas may be obtained by using the apparatus shown in Figs. 130 and 131.

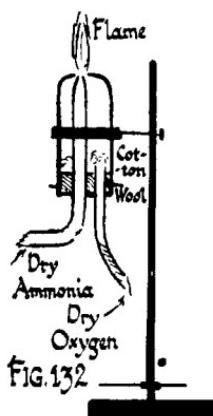
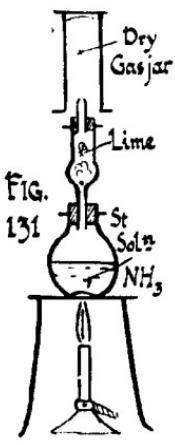
#### PROPERTIES

Very pungent odour, colourless, caustic taste, turns red litmus blue, fumes in contact with hydrochloric acid gas, due to formation of solid ammonium chloride.

Of all gases it is the most soluble in water—the solution is called ammonium hydrate. At ordinary temperatures 1 volume of water will dissolve between 700 and 800 volumes of ammonia. The solution is attended by *considerable increase in volume*, and decrease in density, so that strong ammonia has a sp. gr. of 0·88. Fig. 66, page 18, shows the apparatus usually used to illustrate the great solubility of ammonia in water.

It is a very light gas, being only slightly more than half as heavy as air. It is fairly easily condensed to a liquid which has a considerable industrial application as a refrigerating agent for the production of blocks of ice.

Ammonia does not burn in air, but if it is first mixed with oxygen it will do so, producing a greenish yellow flame. Fig. 132 illustrates a form of apparatus which can be used.



- Ammonia is used for neutralizing acids in dye-stuffs; by dyers when milder alkali than soda is needed or when volatility is desired;
- in the manufacture of Turkey red oil;
- as a fixing agent for certain metallic mordants.

Ammonium chloride is used for aniline blacks, and ammonium carbonate (sal volatile), which may be prepared by heating ammonium sulphate with chalk and collecting the sublimate, is used as a substitute for danting in dyeing Turkey reds.

Ammonium acetate, made by mixing equivalent quantities of ammonia and acetic acid, is used with alizarines, and as a stripping agent for dyed wool and silk.

Ammonium oxalate is sometimes used in wool-dyeing.

## SECTION IX

### THE ELEMENTS OF CHEMICAL THEORY

#### I. THE ATOMIC THEORY

**W**HAT the *ultimate* constitution of matter is, will probably never be thoroughly known; but that does not prevent speculation upon the point. Ideas of this kind are called *theories*, and the one accepted at the present day is that proposed by Dalton, who *adapted* it in 1808 from an ancient Greek idea.

The main principle of it is that *matter is not infinitely divisible*—that ultimately a point is reached beyond which it is impossible to divide up the substance by any physical or chemical operations. We then reach a small particle called the **ATOM**.

There is very little direct proof of the truth of this theory, and it might at any time have to be abandoned, should newly discovered facts oppose it, but at the present time it is *accepted* for many reasons that will appear as the study of chemistry is continued.

By this theory atoms are assumed to be :—

1. *Elementary* in their nature. Therefore there can be only the same number of sorts as there are elements.

2. Endowed with a *mutual attractive force* called chemical affinity or attraction, by virtue of which, when brought into intimate contact, they *combine* with one another. This chemical affinity varies with the kind of atom.

3. Absolutely *indestructible*.

4. (If of the same kind) Of equal mass and alike in all respects.

*Note*.—Nothing is said about the shape of an atom, nor the size. Dalton originally used certain symbols to denote them, such as  $\circ$  for oxygen,  $\ominus$  for nitrogen,  $\odot$  for hydrogen,  $\bullet$  for carbon,  $\oplus$  for sulphur,  $\otimes$  for phosphorus, etc., and hence the idea arose that they were thought to be spherical.

From assumption 2 (above), it is apparent that we shall often have aggregations or groups of atoms. These are known as *Molecules*. Molecules may be made up of atoms *all of the same kind*, in which case

we have *elements*, or *different kinds* of atoms may unite, forming *compounds*.

A molecule may be defined as the smallest mass of any element or compound which can be supposed to exist alone and have all the properties of that element or compound.

As a rule, molecules of elements consist of two or more atoms ; the exceptions are zinc, potassium, sodium, cadmium, mercury (one atom each) ; arsenic, phosphorus (four atoms) ; ozone (three atoms).

Molecules of compounds do not contain a very large number of atoms—seldom more than ten. The exceptions are the compounds of carbon, which sometimes contain hundreds, e.g. starch is thought to have 4,200 atoms per molecule.

The atomic theory's best bulwark is the support given it by the laws of chemical combination, which are completely explained by it ; in fact, it was from a careful study of two of them that Dalton was led to formulate it.

## II. ATOMIC AND MOLECULAR WEIGHTS

We shall here consider what is meant by atomic and molecular weights. The methods by which they may be determined are too difficult for beginners to follow.

As an atom is so small, it is of course utterly impossible to weigh *one* directly ; therefore the mass is given relatively, and originally the number which denoted the atomic weight of an element expressed *the number of times it was as heavy as one atom of hydrogen*.

It was found that one atom of oxygen was nearly sixteen times as heavy as one atom of hydrogen. Now in practice it is much easier to experimentally determine atomic weights with reference to oxygen rather than hydrogen. And if  $\frac{1}{16}$  of the atomic weight of oxygen be considered as unity, the atomic weight of hydrogen becomes 1.008 instead of 1. This system of calculation is the one adopted in the International Table of Atomic Weights.

For all elementary purposes the variation is negligible.

### List of Approximate Atomic Weights of the Commoner Elements

#### *Metals*

Aluminium . . . . .	27	Magnesium . . . . .	24.4
Barium . . . . .	137	Manganese . . . . .	55
Calcium . . . . .	40	Mercury . . . . .	200
Chromium . . . . .	52	Potassium . . . . .	39
Copper . . . . .	64	Silver . . . . .	108
Iron . . . . .	56	Sodium . . . . .	23
Lead . . . . .	207	Tin . . . . .	118.5
Zinc . . . . .	65.5		

<i>Non-metals</i>			
Boron . . . . .	11	Hydrogen . . . . .	1
Bromine . . . . .	80	Iodine . . . . .	127
Carbon . . . . .	12	Nitrogen . . . . .	14
Chlorine . . . . .	35.5	Oxygen . . . . .	16
Fluorine . . . . .	19	Phosphorus . . . . .	31
Sulphur . . . . .			32

By the molecular weight is meant the number of times the molecule of an element or compound is as heavy as one atom of hydrogen or  $\frac{1}{16}$  of one atom of oxygen.

For elements that contain two atoms, this will be twice the atomic weight. For compounds it is the sum of the weights of all the atoms in the molecule.

### III. THE LAWS OF CHEMICAL COMBINATION

Scientific laws are not made—they are discovered. Their application is the opposite to “man-made laws.”

If the individual breaks the law of the land, he is penalized—in other words, the individual has to conform to the law. In science the law has to conform to individual experiment. Should experimental work fail to agree with the law, the law is assumed to be stated wrongly.

The following laws have, so far, never been contradicted by accurate experiment, but have been confirmed in thousands of instances.

**1. Proust's Law of Constant (or fixed, or definite) Proportion.** The same compound always contains the same elements united together in exactly the same proportion by weight.

E.g. calcium carbonate, whether it occurs as chalk, or Iceland spar, or calcite, or marble, or aragonite, or egg shell, or oyster shell, always contains in 100 grams of it 40 of calcium, 12 of carbon, and 48 of oxygen.

Magnesium oxide, whether made by heating magnesium in air or by ignition of the nitrate or carbonate, or in any other way, always consists of 24 parts of magnesium to 16 of oxygen.

**2. Dalton's Law of Multiple Proportions.** When the *same two* elements combine together to form more than one compound, the proportions by weight in the other compounds are always some simple multiple of the proportion found in the simplest compound.

E.g. Hydrogen and oxygen unite to form two compounds, (a) water, in which the ratio of hydrogen to oxygen is 1 to 8, and (b) hydrogen peroxide, in which the ratio is 1 to 16 ( $8 \times 2$ ).

Copper and sulphur unite to form two sulphides in which the proportion of copper and sulphur are respectively 64 : 32 and 128 : 32.

Nitrogen and oxygen form a series of compounds—the oxides of

**nitrogen**—in which the following proportions of nitrogen and oxygen are found :—

Nitrous oxide . . . .	28 : 16, i.e. 28 : 16
Nitric " . . . .	28 : 32 " 28 : 16 × 2
Nitrogen trioxide . . . .	28 : 48 " 28 : 16 × 3
Nitrogen peroxide . . . .	28 : 64 " 28 : 16 × 4
Nitrogen pentoxide . . . .	28 : 80 " 28 : 16 × 5

This phenomenon is quite explicable if we imagine the respective elements “ moving about ” in certain definite entities, such as atoms.

**3. Richter's Law of Reciprocal (or equivalent) Proportions.** When two different elements unite with the same quantity of a third element, the proportions in which they do so will be the same as, or some simple multiple of, the proportions in which they unite with each other.

This is best illustrated by a diagram. Let A, B, C represent respectively three elements. Suppose B combines with A in the

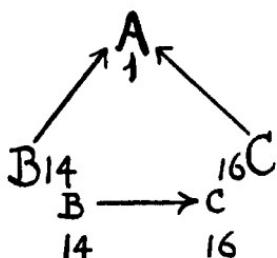


Fig. 133

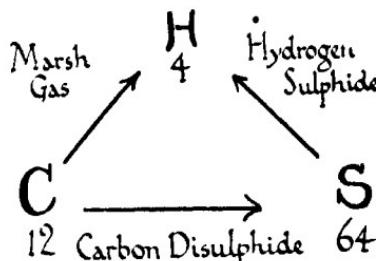


Fig. 134

proportion 14 : 1 ; and suppose C also combines with A in the proportion 16 : 1. (Note same quantity of A.)

Then B will combine with C in the proportion 14 : 16, or some simple multiple of this proportion (Fig. 133), e.g. in *marsh gas* 12 parts of carbon are combined with 4 of hydrogen. In *sulphuretted hydrogen* 64 parts of sulphur are combined with 4 of hydrogen ; and we find in *carbon disulphide* that 12 parts of carbon are combined with 64 of sulphur—which supports the truth of the Law of Reciprocal Proportions (Fig. 134).

#### EXPERIMENTS ILLUSTRATING THE LAWS OF CHEMICAL COMBINATION

1. *To determine the Composition of Magnesium Oxide made in Three Ways.* Weigh very accurately three pieces of bright magnesium ribbon. Each piece should weigh at least 0·1 gram, but not more than 0·2 gram. Treat as follow :

*Piece No. 1.* Put it in a weighed porcelain crucible and heat it over a hot flame until it burns and is completely converted into white magnesium oxide. Prevent the escape of any of the light powder by holding the lid at an angle just above the top of the crucible, using the tongs. Let the vessel cool and then weigh. The increase in weight represents oxygen which has combined with metal to form the oxide.

*Piece No. 2.* Put this in a small weighed evaporating basin, and, after covering with a watch-glass to prevent loss during solution, add two or three drops of dilute nitric acid. Repeat until the metal is dissolved. Put the dish on a water bath, and when all the water has evaporated, heat over a hot flame until the magnesium nitrate is completely converted into the oxide. N.B.—Magnesium nitrate fuses when heated, and decomposes with liberation of nitric oxide to produce infusible white magnesium oxide. Weigh dish and contents, and calculate the proportion of oxygen to magnesium in this sample.

*Piece No. 3.* Put this in a small beaker and again dissolve in nitric acid; dilute this solution with a few c.c. of water and then add sodium carbonate solution to produce magnesium carbonate. Filter to collect the precipitate on filter paper and dry over a small flame.

When dry, transfer it to a weighed crucible, and heat very strongly until the carbonate is again converted into the oxide. N.B.—The carbonate loses weight on heating, the oxide does not.

Carefully tabulate the results you obtain, showing clearly the proportion of oxygen to magnesium in each sample, expressing the quantity as per cent. oxygen and per cent. magnesium in each case.

*2. To determine the Relationship between the Proportions in which Oxygen unites with Lead to form Lead Peroxide and Litharge.*

Weigh a crucible and put in it a few grams of lead peroxide, carefully determining its weight. Heat this gently over a small flame, stirring at intervals with a small piece of iron wire until it is converted into litharge.

Take care that it does not fuse. N.B.—Litharge is buff in colour. Find the loss in weight—which represents the first portion of oxygen liberated.

After weighing add a few pieces of solid potassium cyanide, put the lid on and fuse up the mixture. Ultimately a button of molten lead will be formed in the bottom of the crucible. Cool, wash out all the soluble salts, dry, and weigh. Find the second loss due to oxygen, and compare the two results. N.B.—Potassium cyanide is a deadly poison (a salt of prussic acid); therefore take the greatest precautions in handling it.

*3. Compare the Proportions of Sulphur and Copper in Copper Sulphide.*

Make one sample by dissolving a weighed amount of copper in

nitric acid, and precipitating the sulphide with excess of ammonium sulphide, filtering, drying, and weighing.

Make the other sample from pure precipitated copper by heating gently in a crucible with flowers of sulphur. The cake formed should be broken up several times, and a little more sulphur added each time. Finally, the excess sulphur should be very carefully burnt off with the lid of the crucible removed.

#### IV. THE LAW OF GASEOUS VOLUMES (Gay Lussac)

(which must not be confused with the physical law relating to expansion of gases, known as the Law of Charles or Gay Lussac).

When chemical action takes place between *gases*, either elementary or compound, the volume of the gaseous product bears a *simple* relation to the volumes of the reacting gases, e.g. :—

1 volume of hydrogen unites with 1 volume of chlorine to form 2 volumes of hydrogen chloride.

1 volume of nitrogen unites with 3 volumes of hydrogen to form 2 volumes of ammonia.

1 volume of oxygen unites with 2 volumes of carbon monoxide to form 2 volumes of carbon dioxide.

2 volumes of hydrogen unite with 1 volume of oxygen to form 2 volumes of steam,  
etc.

These are often called *two-volume formula gases*.

A "two-volume formula" is generally written  $\square\square$ . Thus

$\square$  of hydrogen,  $\square$  of chlorine =  $\square\square$  of hydrogen chloride.

$\square$  of nitrogen,  $\square\square$  of hydrogen =  $\square\square$  of ammonia.

By examining these expressions we can arrive at the relationship between the original and resulting volumes of the reacting gases.

N.B.—This notation must *not* be used for solids, e.g. this is *wrong*:

$\square$  of carbon,  $\square\square$  of oxygen =  $\square\square$  of carbon dioxide.

In common language we may sum up the law as follows: "Gases always combine in equal bulk or 1 part by bulk of one with 2 or 3 parts of the other."

#### EXPERIMENTAL METHODS FOR DEMONSTRATING THIS RELATIONSHIP FOR SOME OF THE COMMONER GASES

1. *To show that 2 Volumes of Hydrogen unite with 1 Volume of Oxygen to form 2 Volumes of Steam.*

Fit up the graduated eudiometer tube surrounded with a jacket as shown in Fig. 135. Pass in hydrogen until it fills two graduations, when the pressure is equal in each limb of the eudiometer. Now add oxygen till the third graduation is reached. Circulate steam (or, better, amyl alcohol vapour) until the gases are heated to at least

100° C. and again adjust the pressure. The volume of gas has increased; mark this position *on the outside of the jacket*. Put a screw clip on the rubber tube connecting the eudiometer with the levelling tube, and then "spark" the gas, taking care to first cover the apparatus with a thick duster in case the tube bursts.

Unscrew the clip and adjust the pressure. It will be found that the volume now occupied by the steam will be two-thirds of the

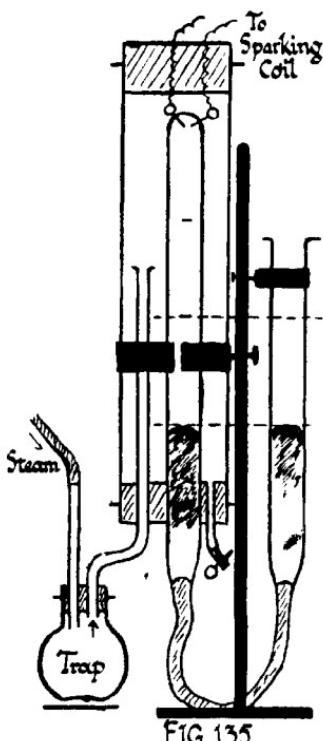


FIG. 135

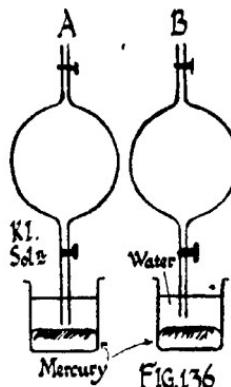


FIG. 136

volume occupied by the mixed gases just before the spark was passed.

2. *To show that 1 Volume of Hydrogen combines with 1 Volume of Chlorine to form 2 Volumes of Hydrogen Chloride.*

For this experiment, two glass bulbs with side tubes are required filled with the mixed gases obtained by the electrolysis of strong hydrochloric acid. It is desirable, although not essential, that each side tube be provided with a glass tap (Fig. 136).

One of them (say A) is wrapped in red paper and put away in a

dark cupboard and the other is exposed to diffused daylight, *but not sunlight*, for several days until all green colour has disappeared. The bulbs are placed over separate vessels containing mercury, into which the end of one of the side tubes is made to dip, and the tap on this tube is opened. It will be found that the result in each case is the same, namely, no gas comes out of the bulb and no mercury passes into it; the volumes of original gas and resulting gas are thus identical.

On the top of the mercury in the vessel under A is put a solution of potassium iodide, and the end of the tube is raised until it reaches this liquid. The solution commences to rise because *it dissolves the free chlorine*. When it exactly half fills the bulb, it stops. If the bulb be now transferred to a deeper vessel, depressed in it, and the top tap opened, the gas expelled will be found to be all hydrogen.

In the vessel under B should be put water, and on bringing the end of its tube in contact with this liquid, it will be found that all the gas is soluble in it, i.e. it is all hydrochloric acid gas. If the capacity of a bulb be called 2 volumes, half the capacity will count as 1 volume, from which we obtain the relationship 1 volume of hydrogen combines with 1 volume of chlorine to form 2 volumes of hydrogen chloride.

### 3. To show that 1 Volume of Nitrogen combines with 3 Volumes of Hydrogen to form 2 Volumes of Ammonia.

For this demonstration we assume the truth of the value obtained in the previous experiment, namely, that hydrogen and chlorine combine in equal volumes.

A tube about a yard in length is required, provided with a well-fitting rubber stopper through which passes a dropping funnel with a well-ground glass tap (Fig. 137).

The stopper is removed and the tube completely filled with chlorine. This is best done over strong brine. The stopper is reinserted and strong ammonia solution put into the funnel. The tap is very carefully turned and a few drops of the liquid allowed to enter the tube.

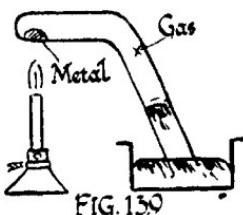
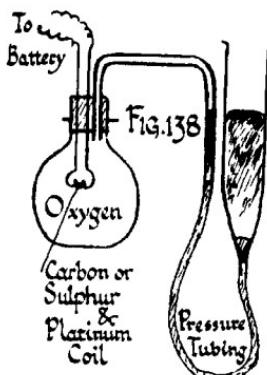
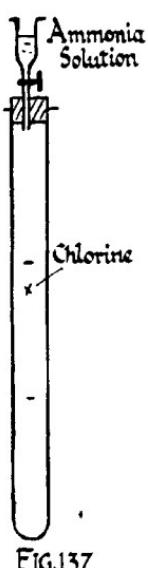
The ammonia is immediately decomposed by the chlorine to form hydrochloric acid with liberation of nitrogen, the energy of combination being sufficient with the first two or three drops to produce flashes of light. A little more ammonia is run in and the tube inverted several times until all the chlorine is used. Dilute sulphuric acid is now put into the funnel and run into the tube. This neutralizes the excess of ammonia, and dissolves the ammonium chloride crystals that have also been formed. Water is then allowed to enter until the tube is full of liquid and gas, which will be so when a bubble of gas attempts to pass up through the funnel.

The gas left in the tube is measured and compared with the volume of chlorine used. The result will be as 1 is to 3. The residual gas can now be proved to be nitrogen, and as 3 volumes of hydrogen were

required to satisfy 3 volumes of chlorine, the ratio of nitrogen to hydrogen in ammonia must be 1 to 3.

4. To show that Carbon Dioxide and Sulphur Dioxide each contains its own Volume of Oxygen.

A small flask is fitted with a tight-fitting rubber stopper through which pass two copper rods and a glass tube. To the ends of the former, which are to be put inside the flask, is attached a spiral of thin platinum wire. The glass tube is bent into the form of a double right-angled bend, for use with a manometric tube (Fig. 138).



A small piece of charcoal or sulphur is put in the spiral, the flask filled with oxygen (by means of a tube not shown in the figure), and the level of mercury in the manometer tube is marked. The ends of the copper rods are connected to the terminals of a suitable battery and a current of electricity is passed through, with the result that the platinum becomes red-hot, which heats the carbon or sulphur sufficiently to make it burn to carbon dioxide or sulphur dioxide.

Due to the heating effect, the volume of gas is increased, but when it is allowed to cool down to the original temperature, it is found that the final volume is the same as the original. In other words carbon dioxide or sulphur dioxide contains its own volume of oxygen.

5. To show that Nitrous Oxide contains an equal Volume of Oxygen, Nitric Oxide half its Volume of Oxygen, and Sulphuretted Hydrogen its own Volume of Hydrogen.

For all these determinations the same piece of apparatus can be used—known as a “thumb tube” (Fig. 139).

It is filled with one of these gases and put over a vessel containing mercury, the level of which is marked on the tube. A small pellet of metal (potassium for the oxides of nitrogen, and tin for sulphuretted hydrogen) is pushed under the surface of the mercury in the dish and allowed to rise to the surface of the mercury in the tube.

The finger is then put under the end of the tube and the pellet jerked into the horizontal portion. This is then carefully heated. The potassium combines with the oxygen in the oxides of nitrogen, liberating nitrogen; and the tin decomposes the sulphuretted hydrogen, forming tin sulphide and liberating hydrogen.

The gas is allowed to cool and the level of mercury compared with the original, from which the ratios will be found to be as that stated above.

## V. AVOGADRO'S LAW

states that “equal volumes of all gases under the same conditions of temperature and pressure contain the same number of molecules.”

This law is now admitted to be as true as the other four, but its proof is beyond the requirements of a student of elementary chemistry, as it requires more than an elementary knowledge of mathematics and physics.

At this stage it must be accepted.

## EQUIVALENTS

When chemical reaction occurs between certain substances, notably acids and metals, it is found that one element has usually replaced another element in one of the compounds present.

Further, a certain mass of one element is found to displace consistently the same quantity of the other element. These two quantities are said to be *equivalent*.

It is convenient in practice to adopt some *one* element to which all the others refer, and thus the *equivalent of an element* is defined as the number of parts (or grams) of an element which are replaceable by, or combine with, or are chemically equal to, one part (or gram) of hydrogen.

## EXERCISES IN DETERMINATION OF EQUIVALENTS

As in a great many of these determinations a volume of hydrogen will be collected, which must be converted into a mass, the first exer-

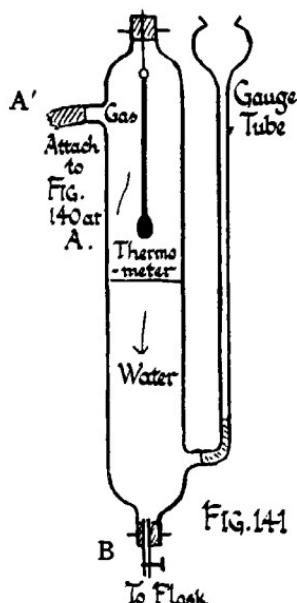
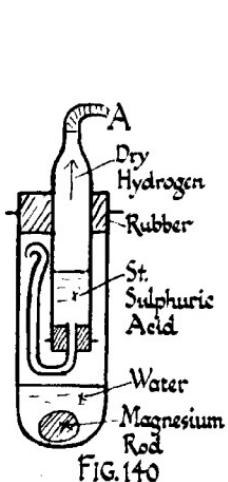
oise is to determine the relationship which exists between these two quantities.

*1. To determine the Mass of 1 Litre of Hydrogen.*

Use the special apparatus, diagrams of which are shown in Figs. 140 and 141.

Carefully weigh the light apparatus (Fig. 140) when fitted up with a *small* piece of magnesium rod in the T.T., covered with water, and strong sulphuric acid partly filling the drying bulb.

Attach it to the collecting apparatus A to A'; arrange the level of water in aspirator and gauge tube to be the same by drawing off water at B.



Gently warm the T.T. Air is expelled through acid. Cool. Acid is drawn into outer tube, and hydrogen is liberated, passes through the drying bulb and into the aspirator, causing the water in gauge tube gradually to rise. Draw off water at B into a 250 c.c. flask at such a rate as will *keep the surfaces of the water level*.

When *all* action is over

(1) Read thermometer in aspirator (say  $t^{\circ}$  C.).

(2) Read atmospheric pressure on barometer (say P mm.).

(3) Detach the light apparatus and carefully weigh to find the

*loss* in mass, which gives the *mass* of hydrogen evolved (say =  $m$  grams).

(4) Carefully find the *volume* of water collected by filling up the 250 c.c. flask from a burette—which gives the *volume* of hydrogen evolved (say =  $a$  c.c.).

Volume corrected to N.T.P. (i.e.  $0^{\circ}$  C. and 760 mm. pressure)

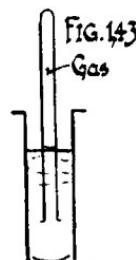
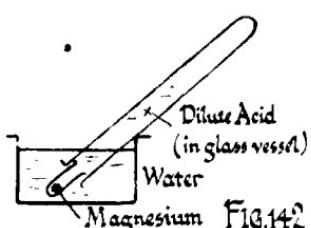
$$= \frac{a}{1 + (0.00367 \times t)} \times \frac{P}{760} \text{ [by Boyle's and Charles's law].}$$

This has a mass of  $m$  grams.

$$\therefore 1 \text{ litre weighs } \frac{m}{\frac{a}{1 + (0.00367 \times t)} \times \frac{P}{760}} \times \frac{1,000}{1} \text{ grams.}$$

Correct value = .0899 gram.

2. To determine the Equivalent of a Metal by dissolving in Acid and collecting the evolved Hydrogen.



Several variations of apparatus are given according to the metal to be used.

(a) *Magnesium*. Weigh out very accurately about 1 gram of freshly cleaned magnesium ribbon. Put it into the bottom of a narrow T.T., and fill with water by dropping into a small glass or enamelled-iron pneumatic trough.

Fill the larger tube—half with dilute sulphuric acid and half with water—close with thumb, place in trough, slip in the T.T. (Fig. 142) and raise to vertical position.

When the magnesium is all dissolved, take the temperature of the water and transfer the tube carefully to a tall cylinder of water to bring to atmospheric pressure, which is equal to the barometric pressure (Fig. 143).

Mark the level with a strip of paper, and find the volume of the gas by pouring in water from a measuring vessel.

Correct this volume to  $0^{\circ}$  C. and 760 mm. pressure, and convert into a mass (in grams) by means of the value found in Exercise 1.

By "Rule of Three," find

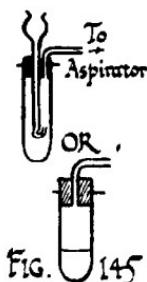
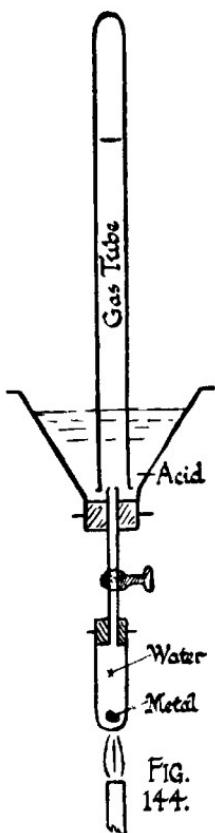
*How many grams of magnesium would be required in order to liberate 1 gram of hydrogen (i.e. the equivalent of magnesium).*

The apparatus shown in Figs. 144 and 145 can also be used.

(b) Zinc, Iron, Aluminium. Use the apparatus shown in Fig. 144.

Carefully weigh a small piece of metal and place in T.T. Fill it with water, and close the tap.

Place dilute acid in the funnel, fill the collecting tube, invert it into the funnel, and open the tap.



The acid flows down and ultimately acts upon the metal—the gas evolved being collected in the closed tube.

The temperature of the gas must be determined in each case, and the volume corrected to *atmospheric* pressure as shown in Fig. 143.

Correct the volume and find its mass as in (a) above.

For zinc and iron, use either dil. sulphuric or dil. hydrochloric acid.

For aluminium use either caustic soda solution or hydrochloric acid (1 volume water, 1 volume strong acid).

It is desirable, and in some cases necessary, to warm gently.

Apparatus shown in Fig. 145 is to be used with aluminium and caustic soda.

Be careful that the stoppers fit very tightly and are of *rubber*.

Calculate how many grams of the given metal are required to liberate 1 gram of hydrogen.

*3. To determine the Equivalent of a Metal by Precipitation with another whose Equivalent has been already determined.*

(a) Prepare a solution in water of a soluble salt of the metal whose equivalent is to be determined, e.g.

For *copper*, use copper sulphate.

„ *silver*, „ silver nitrate.

„ *tin*, „ stannous chloride in hydrochloric acid.

„ *lead*, „ lead acetate.

(b) Carefully weigh a quantity of the precipitating metal, which must be as pure as possible.

*Zinc* (foil) will precipitate copper, silver, tin, lead.

*Copper* (foil) will precipitate silver (in hot solution).

*Magnesium* will precipitate silver, iron, cobalt, zinc, nickel, gold, platinum, bismuth, tin, mercury, copper, lead, cadmium.

*Iron* will precipitate copper.

Add the precipitating metal to the solution contained in a *small beaker*, stir from time to time with a glass rod, until all the precipitating metal is dissolved.

Collect every particle of the precipitated metal, either on a filter paper (of known mass) or in a small crucible; wash well first with hot distilled water, then with alcohol; and dry in a steam oven. When completely dry, weigh.

Finally, calculate the equivalent of one metal in terms of the other; and then, to obtain the equivalent in reference to hydrogen, multiply this result by the equivalent of the known metal.

*4. To determine the Equivalent of an Element by preparing a Compound of the Element with another whose Equivalent has been previously determined.*

(a) *Oxygen.* (i) Prepare tin oxide. Use a known mass of granulated tin.

Place it in a crucible and add strong nitric acid from a pipette drop by drop (Fig. 146). When all the tin is oxidized, ignite and weigh.

(1) Calculate what mass of oxygen has combined with the equivalent weight of tin.

(2) Prepare magnesium oxide by the action of dilute nitric acid on a known mass of the metal, and perform similar calculations.

(b) *Silver.* Prepare pure silver oxide by allowing baryta water to filter into a small flask of silver nitrate solution. Cork at once and shake; allow the oxide to settle; decant liquid; wash several times with hot water, and dry in steam oven. Put two or three grams into a hard glass tube of known mass. Attach to an aspirator, heat, and collect the evolved oxygen. Find its mass—check by weighing the residue in the tube.

Calculate the amount of silver which combines with eight grams of oxygen.

Table of Approximate Equivalents

Hydrogen	= 1	Sodium	= 23
Carbon	= 3	Iron	= 28
Nitrogen	= 4.7	Copper	= 32
Oxygen	= 8	Zinc	= 32.7
Aluminium	= 9	Chlorine	= 35.5
Magnesium	= 12.2	Tin	= 59
Sulphur	= 16	Silver	= 108

Equivalence is an experimental fact and shows no variation—except that certain elements (that form two series of salts) give one or other of two values, depending upon which class of compound is being experimented with, e.g.

Mercury 100 or 200. Copper 32 or 64.

Tin 59 or 118. Iron 18.67 or 28.

These values are related to each other as 1 : 2, 2 : 3, etc.

#### THE THEORY OF VALENCY

is an attempt to *explain*, chiefly from logical considerations, the phenomenon of equivalency. It is found that if the atomic or combining weight of an element be divided by its equivalent, in *every instance* the quotient is a whole number (1, 2, 3, 4, 5). These numbers are *assumed* to measure the ratio of chemical combining powers of the respective elements.

Table of Valencies or Atomicities

Monads.	Diads.	Triads.	Tetrad.	Pentads.
Chlorine	Calcium	Aluminium	Carbon	Nitrogen
Hydrogen	Copper	Nitrogen	Tin	Phosphorus
Potassium	Iron	Phosphorus		
Silver	Zinc			
Sodium	Lead			
	Magnesium			
	Oxygen			
	Sulphur			
	Tin			

Those giving a quotient of 1 are termed monads or monovalent elements.

Those giving a quotient of 2 are termed diads or divalent elements.

Those giving a quotient of 3 are termed triads or trivalent elements.

The relationship existing among atomic weight, equivalence, and valency may be summarized as :—

$$\text{Atomic weight} \div \text{Valency} = \text{Equivalent or } E = \frac{\text{At. Wt.}}{V}$$

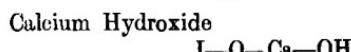
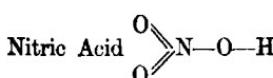
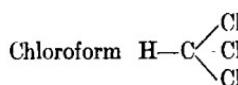
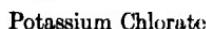
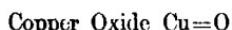
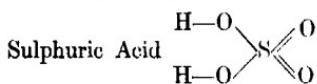
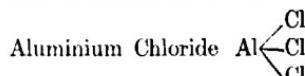
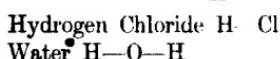
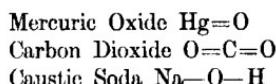
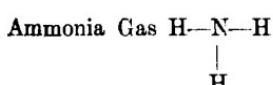
and as in most cases At. Wt. = Molecular Wt.  $\div$  2 and Molecular Wt. = Vapour Density  $\times$  2,

Therefore

$$V = \frac{\text{v.d.}}{E}.$$

Thus if the vapour density of an element = 16 and its equivalent is 8, its valency is  $\frac{16}{8} = 2$  (i.e. it is divalent).

#### GRAPHIC REPRESENTATION OF VALENCY



#### SYMBOLS, FORMULÆ AND EQUATIONS

Symbols were devised by Dalton to represent one atom of the respective elements. He used circles, e.g. O, O, ⊖, ⊕, for certain of the non-metallic elements, and circles containing letters for the metals. Berzelius brought symbolic representation to its present form by omitting the circles and using initial letters throughout, e.g.:—

Aluminium . . . . .	Al	Hydrogen . . . . .	H
Chlorine . . . . .	Cl	Magnesium . . . . .	Mg
Iron (Ferrum) . . . . .	Fe	Oxygen . . . . .	O
Mercury (Hydrargyrum) . . . . .	Hg	Potassium (Kalium) . . . . .	K
Silver (Argentum) . . . . .	Ag	Carbon . . . . .	C
Phosphorus . . . . .	P	Iodine . . . . .	I
Barium . . . . .	Ba	Nitrogen . . . . .	N
Copper (Cuprum) . . . . .	Cu	Sulphur . . . . .	S
Lead (Plumbum) . . . . .	Pb	Zinc . . . . .	Zn
Sodium (Natrium) . . . . .	Na		etc.
Calcium . . . . .	Ca		

The symbol denotes :—

1. One atom of the element.
2. The number of parts by weight given as its atomic or combining weight.

*It is not to be used as an abbreviation for the name nor as chemical shorthand.*

Combinations of symbols are termed *Formulae*. They (usually) represent the proportions by weight and the number of atoms in which the elements exist in one molecule of the compound.

If a multiple of the atomic weight is denoted, this multiple is written as an index figure to the symbol at the bottom, e.g. H<sub>2</sub>, O<sub>3</sub>, P<sub>4</sub>, Cl<sub>2</sub>. A bracket has the same significance as in arithmetic, e.g. (COOH)<sub>2</sub> means twice all the symbolic value in the bracket.

A figure in front of an expression has the same value as before a bracket, and carries its influence to the end of the expression.

7H<sub>2</sub>O means seven times the sum of H<sub>2</sub> and O.

The formula of a substance represents one molecule of that substance.

#### Formulae of some Common Compounds

Hydrochloric Acid . . . . .	HCl	Copper Sulphate . . . . .	CuSO <sub>4</sub> .5H <sub>2</sub> O
Sodium Chloride . . . . .	NaCl	Ferrous Sulphate . . . . .	FeSO <sub>4</sub> .7H <sub>2</sub> O
Calcium Chloride . . . . .	CaCl <sub>2</sub>	Magnesium Sulphate . . . . .	MgSO <sub>4</sub> .7H <sub>2</sub> O
Potassium Chloride . . . . .	KCl	Zinc Sulphate . . . . .	ZnSO <sub>4</sub> .7H <sub>2</sub> O
Ammonium Chloride . . . . .	NH <sub>4</sub> Cl	Barium Sulphate . . . . .	BaSO <sub>4</sub>
Ferric Chloride . . . . .	Fe <sub>2</sub> Cl <sub>3</sub>	Calcium Sulphate . . . . .	CaSO <sub>4</sub>
Silver Chloride . . . . .	AgCl	Ammonium Sulphate . . . . .	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>
Lead Chloride . . . . .	PbCl <sub>2</sub>	Sodium Sulphate . . . . .	Na <sub>2</sub> SO <sub>4</sub>
Magnesium Chloride . . . . .	MgCl <sub>2</sub>	Potassium Sulphate . . . . .	K <sub>2</sub> SO <sub>4</sub>
Zinc Chloride . . . . .	ZnCl <sub>2</sub>	Carbon Dioxide . . . . .	CO <sub>2</sub>
Nitric Acid . . . . .	HNO <sub>3</sub>	Calcium Carbonate . . . . .	CaCO <sub>3</sub>
Silver Nitrate . . . . .	AgNO <sub>3</sub>	Sodium Carbonate . . . . .	Na <sub>2</sub> CO <sub>3</sub>
Sodium Nitrate . . . . .	NaNO <sub>3</sub>	Sodium Bicarbonate . . . . .	NaHCO <sub>3</sub>
Potassium Nitrate . . . . .	KNO <sub>3</sub>	Calcium Bicarb. . . . .	CaH <sub>2</sub> (CO <sub>3</sub> ) <sub>2</sub>
Copper Nitrate . . . . .	Cu(NO <sub>3</sub> ) <sub>2</sub>	Ammonium Carbonate . . . . .	(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>
Sulphuric Acid . . . . .	H <sub>2</sub> SO <sub>4</sub>	K <sub>2</sub> Titanium Chlorate . . . . .	KClO <sub>3</sub>

## THE ELEMENTS OF CHEMICAL THEORY

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Potassium Permanganate	K <sub>2</sub> Mn <sub>3</sub> O <sub>8</sub>	Mercuric Oxide	HgO
Chloroform	CHCl <sub>3</sub>	Zinc Oxide	ZnO
Water	H <sub>2</sub> O	Ozone	O <sub>3</sub>
Carbon Monoxide	CO	Sodium Hydrate	NaOH
Sulphur Dioxide	SO <sub>2</sub>	Calcium Hydrate	Ca(OH) <sub>2</sub>
Silicon Dioxide (sand)	SiO <sub>2</sub>	Barium Hydrate	Ba(OH) <sub>2</sub>
Manganese Dioxide.	MnO <sub>2</sub>	Copper Hydrate	Cu(OH) <sub>2</sub>
Magnesium Oxide	MgO	Potassium Hydrate	KOH
Sulphur Trioxide	SO <sub>3</sub>	Hydrogen Sulphide	H <sub>2</sub> S
Copper Oxide	CuO	Carbon Disulphide	CS <sub>2</sub>
Nitrous Oxide	N <sub>2</sub> O	Ferrous Sulphide	FeS
Nitric Oxide	NO	Ammonia	NH <sub>3</sub>
Barium Peroxide	BaO <sub>2</sub>	Ammonium Hydrate	NH <sub>4</sub> OH
Litharge	PbO	Mercuric Iodide	HgI
Red Lead	Pb <sub>2</sub> O <sub>4</sub>	Alum	Al <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> .K <sub>2</sub> SO <sub>4</sub> .24H <sub>2</sub> O
Lead Peroxide	PbO <sub>2</sub>	Bleaching Powder	Ca(OCl)Cl
Ferric Oxide	Fe <sub>2</sub> O <sub>3</sub>	Oxalic Acid	(COOH) <sub>2</sub>
Calcium Oxide	CaO	(Ethyl) Alcohol	C <sub>2</sub> H <sub>5</sub> .OH

## AN EQUATION

represents quantitatively a chemical change or reaction.

+ on the left means "chemically reacting with."

+ on the right means "and."

= or → means "produces, forms, yields," etc.

An equation always contains at least two formulæ. The only points of equality are:

(1) That the sum of the combining weights on one side must equal the sum of the combining weights on the other.

(2) The number of atoms of each element must be the same on each side.

To solve an arithmetical example an equation should be used in the following manner:

**Step 1.** Select the correct equation.

2. Enter the respective combining weights.

3. Total them up for each formula.

,, 4. Underline the terms required.

„ 5. Get the statement for "rule of three."

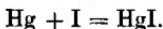
### EXAMPLE

"How much copper would be required to produce 8 grams of copper nitrate by the action of nitric acid on the metal?"

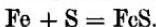
$$\text{Step 1. } 3\text{Cu} + 8\text{HNO}_3 = 3\text{Cu}(\text{NO}_3)_2 + 4\text{H}_2\text{O} + 2\text{NO}$$

,, 2.	$3 \times 64$	$\begin{array}{r} 1 \\ 14 \\ \hline 48 \\ \hline 63 \times 8 \end{array}$	$\begin{array}{r} 14 \\ 3 \times 16 \\ \hline 124 \\ 64 \end{array}$	$\begin{array}{r} \times 2 \\ \hline 2 \\ 16 \\ 30 \times 2 \\ \hline 18 \times 4 \end{array}$	$\begin{array}{r} 14 \\ 16 \\ 30 \\ \hline 18 \end{array}$
,, 3.	$192$	$504$	$564$	$72$	$60$
,, 4.	$=$		$\overline{0}$		

## **Equations representing some well-known Chemical Reactions**



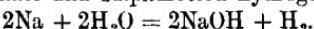
Mercury and iodine combining to form mercuric iodide.



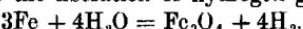
### Iron and sulphur combining to form ferrous sulphide.



Ferrous sulphide and dil. sulphuric acid reacting to produce ferrous sulphate and sulphuretted hydrogen gas.



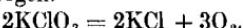
Sodium acting on water to produce sodium hydrate (caustic soda) with the liberation of hydrogen gas.



Iron heated in a current of steam—black oxide of iron formed and hydrogen liberated.



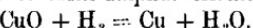
Zinc and dil. sulphuric acid react to produce zinc sulphate and hydrogen.



Potassium chloride is decomposed by heat into oxygen and potassium chloride.



Sulphur dioxide and oxygen passed over heated platinized asbestos unite to form sulphur trioxide.



Copper oxide heated in a current of hydrogen is reduced to metallic copper, with formation of water.



Calcium oxide (lime) exposed to carbon dioxide combines to form chalk.

The following equations represent chemical changes that have taken place while performing some simple experiments in the laboratory. Write in words the meaning of each in a similar manner to that shown above.

- $$\begin{array}{ll} \text{1. } \text{Mg} + \text{O} = \text{MgO} & \text{5. } \text{Zn} + 2\text{HCl} = \text{ZnCl}_2 + \text{H}_2 \\ \text{2. } \text{MnO}_2 + 4\text{HCl} & \text{6. } \text{Fe} + \text{H}_2\text{SO}_4 = \text{FeSO}_4 + \text{H}_2 \\ & \text{= MnCl}_2 + 2\text{H}_2\text{O} + \text{Cl}_2 \\ \text{3. } \text{CuO} + \text{H}_2\text{SO}_4 = \text{CuSO}_4 + \text{H}_2\text{O} & \text{7. } \text{CuSO}_4 + \text{Fe} = \text{FeSO}_4 + \text{Cu} \\ \text{4. } \text{C} + \text{O}_2 = \text{CO}_2 & \text{8. } 2\text{HgO} = 2\text{Hg} + \text{O}_2 \\ & \text{9. } \text{HCl} + \text{NH}_3 = \text{NH}_4\text{Cl} \end{array}$$

10.  $\text{CaCO}_3 + 2\text{HCl} = \text{CaCl}_2 + \text{H}_2\text{O} + \text{CO}_2$
11.  $\text{HCl} + \text{NaOH} = \text{NaCl} + \text{H}_2\text{O}$
12.  $\text{Na}_2\text{CO}_3 + 2\text{HCl} = 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2$
13.  $\text{SO}_2 + \text{O} + \text{H}_2\text{O} = \text{H}_2\text{SO}_4$
14.  $2\text{H}_2\text{O} = 2\text{H}_2 + \text{O}_2$
15.  $3\text{Fe} + 4\text{H}_2\text{O} = \text{Fe}_3\text{O}_4 + 4\text{H}_2$
16.  $\text{Mg} + \text{H}_2\text{SO}_4 = \text{MgSO}_4 + \text{H}_2$
17.  $\text{CO} + \text{O} = \text{CO}_2$
18.  $\text{Na}_2\text{S} + 2\text{HCl} = \text{H}_2\text{S} + 2\text{NaCl}$
19.  $\text{Zn} + \text{CuSO}_4 = \text{ZnSO}_4 + \text{Cu}$
20.  $\text{H}_2\text{SO}_4 = \text{H}_2\text{O} + \text{SO}_2 + \text{O}$
21.  $\text{P}_4 + 5\text{O}_2 = 2\text{P}_2\text{O}_5$
22.  $2\text{Fe}_2 + 3\text{O}_2 = 2\text{Fe}_3\text{O}_4$
23.  $\text{Hg} + \text{O} = \text{HgO}$
24.  $\text{KOH} + \text{HNO}_3 = \text{KNO}_3 + \text{H}_2\text{O}$
25.  $\text{CaCl}_2 + 2\text{AgNO}_3 = 2\text{AgCl} + \text{Ca}(\text{NO}_3)_2$
26.  $\text{NH}_4\text{Cl} + \text{NaOH} = \text{NH}_3 + \text{NaCl} + \text{H}_2\text{O}$

## EXERCISES IN CHEMICAL ARITHMETIC

- How much oxygen can be obtained by igniting 20 grams of potassium chlorate ?
- For how long must steam be passed over red-hot iron at the rate of 2 grams per minute, in order to produce 8 grams of hydrogen ?
- How much ammonium chloride is required in order to prepare 10 grams of ammonia from a mixture of ammonium chloride and lime ?
- How much chlorine could be obtained by acting on 5·6 grams of manganese dioxide with excess of hydrochloric acid and warming the mixture ?
- How much rock salt would it be necessary to use in order to prepare a 20 per cent. solution of hydrochloric acid in water, using 100 grams of water ?
- How much water can you obtain by reducing 25 grams of copper oxide with hydrogen ?
- How much carbon is required to reduce 165 grams of carbon dioxide to carbon monoxide ?
- How much sulphur is there in 268·3 grams of crystallized zinc sulphate ?
- How much zinc is required to precipitate 100 grams of lead from a solution of its nitrate ?
- How many lb. of nitrogen are contained in 1 ton of ammonium sulphate and sodium nitrate, respectively ?
- How much carbon is there in 1 kilogram of cane sugar ?

## RELATIONSHIP BETWEEN THE VOLUME AND MASS OF GASEOUS ELEMENTS AND COMPOUNDS

If *equal volumes* of different gases (under the same conditions of temperature and pressure) be accurately and carefully weighed, it is found that their weights vary (Section VI, page 47). The lightest is hydrogen, 1 litre of which at N.T.P. weighs 0·0899 gram—sometimes called 1 *cubic centimetre*. Oxygen weighs 16 times as much, ammonia 8·5 times, hydrogen chloride 18·25 times, etc.

This relative density of a gas in relation to hydrogen is called its *Vapour Density*. (See Table, page 47.)

Now, Avogadro's Law states that these litres all contain the *same*

*number of molecules.* Therefore the same ratio (i.e. v.d.) gives the relative weights of the respective molecules of gas.

Then, as we have thus obtained the weight of each molecule of gas in terms of the weight of *one molecule of hydrogen*, the question comes, "What is the weight of 1 molecule of hydrogen in terms of our unit, i.e. the weight of 1 atom of hydrogen?" Careful chemical research has answered this as "*two.*" Therefore the molecular weights of gases are obtained by multiplying the v.d. by 2.

Now, if 1 litre of hydrogen at N.T.P. weighs .0899 gram, then 22.4 litres of hydrogen will weigh 2.02 grams, i.e. twice the atomic weight (1.01) of hydrogen.

Thus 22.4 litres of any elementary or compound gas at N.T.P. will weigh its molecular weight in grams.

#### EXERCISES

1. Find the volume of hydrogen (at N.T.P.) which would be evolved by treating 10 grams of zinc with hydrochloric acid.
2. What volume of oxygen at N.T.P. would be produced by strongly heating 5 grams of potassium chlorate?
3. What weight of common salt would be required to furnish sufficient hydrogen chloride to neutralize 100 grams of a 30 per cent. solution of caustic soda? What would be the volume of the gas at N.T.P.?

## SECTION X

### CARBON AND SOME OF ITS COMPOUNDS

**C**ARBON is the most wonderful of all elements. In the elementary condition it occurs as diamond and graphite. Combined with other elements like hydrogen, oxygen, and nitrogen, it forms thousands of compounds which are essential to life and vital processes generally.

From many of these compounds carbon can be prepared in an amorphous condition, e.g. charcoal, lampblack, gas carbon, etc. The phenomenon of an element occurring in different physical conditions is termed allotropy, and the varieties are known as allotropic modifications.

**Diamonds** are octahedral crystals of high sp. gr. (3.5), are extremely hard, and highly refractive to light. These two properties are mainly responsible for the two chief uses, i.e. for rock boring and cutting, and use as a gem.

Lavoisier showed by burning a diamond in oxygen—when carbon dioxide was produced—that it contained carbon, and later Davy showed that it was pure carbon.

**Graphite**, also known as black-lead or plumbago, is a greyish black solid with a distinct lustre, and occurs very widely distributed throughout the world, i.e. Iceland, Siberia, Ceylon, Canada, etc. It is now produced in large quantities artificially at Niagara by the Acheson process. It is used as (1) a lubricant, (2) a polishing medium for shot, ironwork, gunpowder, etc., (3) an ingredient in the “lead” of pencils, (4) a film for electrotyping, (5) an ingredient in plumbago crucibles.

It is soft and soapy in texture, and is attacked when warmed with a mixture of potassium chlorate and nitric acid.

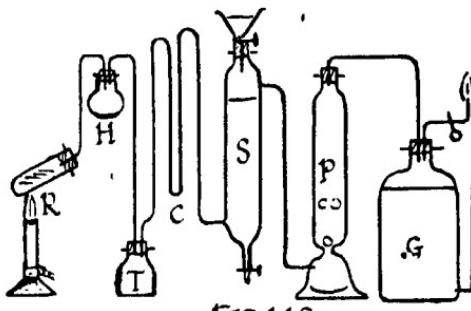
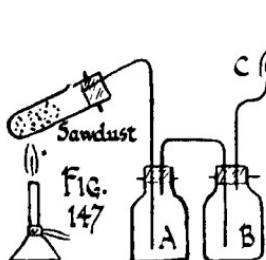
**Amorphous carbon** occurs in many forms, e.g. charcoal, gas, carbon, lampblack, and may be prepared from almost any organic tissue or substance—wood, starch, cheese, sugar, coal, turpentine, etc.

The older style of charcoal production was to make heaps of wood, cover with sods, and set the mass on fire. This was extremely wasteful, and when wood is carbonized to-day it is destructively distilled in ovens in a similar way to that adopted for coal, the result being that

many valuable by-products are obtained of which pyroligneous acid (used as a source of acetic acid), methyl alcohol, and acetone are the most important.

The destructive distillation of wood can be illustrated on a small scale by using the apparatus shown in Fig. 147. The test tube contains sawdust. In bottle A some of the most easily condensed products are obtained. Bottle B, containing water, serves to wash the gas free from others, and an inflammable gas passes along, which may be ignited at C.

**Lampblack** is prepared by burning oil in a small supply of air. A very smoky flame is produced, due to the presence of finely divided carbon. This soot is collected on blankets hung in chambers through which the smoke is made to pass, a process similar to the collection in chimneys. Lampblack is used for printer's ink and black paints. *It will not bleach.*



**Charcoal** of a much purer quality than that obtained by the destructive distillation of wood is prepared by adding strong sulphuric acid to a concentrated solution of sugar in water. The black mass produced is washed free from acid and dried.

**Coke and gas carbon** are obtained in the destructive distillation of coal. The former contains about 90 per cent. of carbon, the latter nearly 100 per cent.

**Animal charcoal** is prepared by roasting bones. Its average composition is 10 per cent. carbon, 38 per cent. calcium phosphate, and 2 per cent. other substances.

It has a very considerable application in industry as a deodorizer and decolorizer, but it requires frequent re-ignition if its efficiency is to remain unimpaired.

**Coal**, to the large deposits of which in this country England largely owes her commercial supremacy, is a very impure form of

carbon, and includes compounds of carbon with hydrogen, nitrogen, etc. It is a mixture evidently formed from a geological deposit of ancient luxurious tropical vegetation.

It is the chief British fuel, and the source of one of the chief means of artificial illumination. For the latter purpose it is destructively distilled to produce "coal gas." Fig. 148 shows the principle of the method.

R represents the retort containing the coal, H the hydraulic main, T the tar-pit, C the coolers, S the scrubber for removing by solution the ammonia produced, P the purifier (containing iron oxide or lime), and G the gasometer.

**Coal tar**, which consists of a mixture of a very large number of compounds of carbon, is also destructively distilled, and the products, as they are evolved at different temperatures, are collected in several fractions. The residuum is known as pitch.

From these fractions chemicals are obtained which severally serve as the starting-points in the manufacture of a series of "intermediate products," these in their turn being used for the preparation of :—

(a) Coal-tar dyes; (b) flavouring, sweetening, and colouring materials; (c) drugs and disinfectants; (d) perfumes; (e) explosives; (f) organic solvents.

*The chief compounds of carbon* include those with :—

1. Oxygen. The OXIDES.—Carbon monoxide, carbon dioxide.
2. Hydrogen. The HYDROCARBONS.—Marsh gas, acetylene, ethylene, benzene, toluene, naphthalene, anthracene.
3. Oxygen and hydrogen. The CARBOHYDRATES (starches, sugars), ALCOHOLS, ALDEHYDES, ETHERS, ACIDS.
4. A metal and oxygen. The CARBONATES, such as chalk, marble, aragonite, witherite, ironstone, soda ash, magnesite, etc.

A few of these compounds will now be considered in detail.

**Carbon dioxide.** A heavy, colourless gas is found to be produced as a result of combustion, respiration, and fermentation, which has been known at various times under various names, e.g. gas sylvestre, choke-damp or after-damp, fixed air, chalk gas, carbonic acid gas, carbonic anhydride, but which is usually termed carbon dioxide.

It is present in fresh air, mixed fairly uniformly, to the extent of 0·03 per cent. to 0·04 per cent. by volume, and is found in certain natural mineral waters, e.g. Apollinaris, Johannis, Perrier, Apenta. It also exudes from vents and fissures in volcanic regions.

Its presence in air is due almost entirely to respiration and combustion. From it it is assimilated by plants and vegetation generally, being converted in the "leaf laboratories" into starch and sugar.

The earth crust contains many carbonates, which are metallic oxides in combination with the gas.

*To prepare the Gas :—*

1. Act on a carbonate with acid, e.g. marble and dilute hydrochloric, magnesite and dilute nitric, sodium carbonate and sulphuric (Figs. 149, 150).
2. Heat charcoal in a good supply of air or oxygen (Fig. 151).
3. Ignite certain carbonates, e.g. chalk, magnesite.
4. Ferment sugar with yeast (Fig. 152), keeping the temperature about 30° C. Enormous quantities of carbon dioxide are obtained

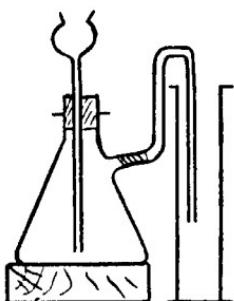


FIG. 149

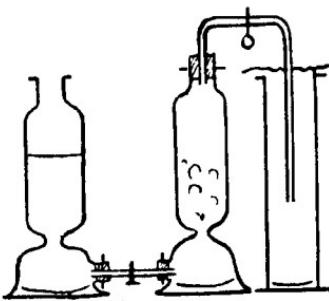


FIG. 150

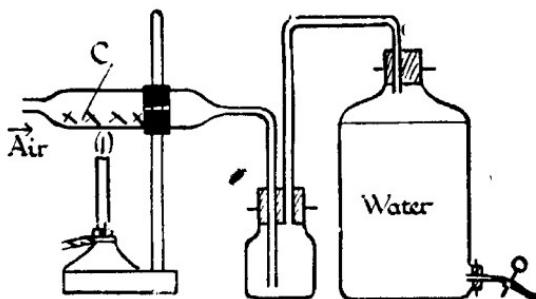


FIG. 151

in this way from breweries and distilleries, condensed into bottles, and used in the manufacture of mineral waters.

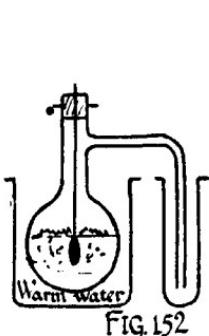
#### PROPERTIES

It is a heavy colourless gas ( $1\frac{1}{2}$  times as heavy as air). Many experiments may be performed to illustrate this property, e.g. a beaker full of it may be weighed and compared with the weight of the same beaker when full of air; it may be poured from one gas jar to another like a liquid; a soap bubble that would fall in air may be floated in a bell jar of the gas (Fig. 153).

The soap bubble is best blown by using a piece of glass tubing about  $\frac{1}{2}$  inch diameter, which contains a plug of cork in which "gutters" have been cut.

It will not support ordinary combustion, e.g. a taper, a candle flame, and even that from burning turpentine will be extinguished by it; but it will support the combustion of burning magnesium, with the liberation of carbon and the formation of magnesium oxide (black and white residue).

It is very soluble in caustic soda, forming sodium carbonate. It is soluble in water, especially under pressure. Soda-water, beer, lemonade, champagne contain it, and thereby "sparkle" when opened.



It is easily liquefied. "Sparklets" contain liquid carbon dioxide. It has a feeble taste, slightly acid reaction when dissolved in water, and a pleasant smell.

- Uses.**
- 1. For making mineral waters.
- 2. For extinguishing fires—hand grenades.
- 3. As a refrigerating agent—liquid carbon dioxide plants are very efficient.
- 4. For aeration of bread and pastry—baking powder liberates it.
- 5. As an asphyxiating agent—the lethal chamber at the famous Lost Dogs' Home in Battersea is worked with it.
- 6. For medicine—Seidlitz powders, health salts, fruit salts, when mixed with water, liberate it.
- 7. As an indicator of impurities in an atmosphere—e.g. in mills and schools.
- 8. As food for plants.

## PRACTICAL EXERCISES

1. *Test for Carbonates.* Use specimens of each of the following carbonates : Calcium carbonate, sodium carbonate, barium carbonate, ammonium carbonate. Test the action of dilute acid on each. Note if carbon dioxide is evolved. Use the apparatus shown in Fig. 154.

Then determine which of the following substances are carbonates or contain carbonates : Iceland spar, egg shell, washing soda, magnesite, oyster shell, oxalic acid, cryolite, baking powder, old mortar.

2. *To distinguish Chalk from Quicklime :—*

(a) Add water, and note result—solubility, temperature.

(b) Try effect of heat on each, and note if any alteration occurs in weight.

(c) Try effect of dilute hydrochloric acid on each.

3. Experiment with the by-product from the manufacture of carbon dioxide from marble and hydrochloric acid, which is calcium chloride.

Evaporate the liquid to dryness, abstract the solid calcium chloride, and describe its colour, texture, action on exposure to air.

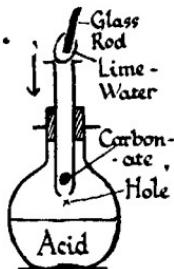


FIG. 154

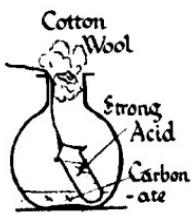


FIG. 155

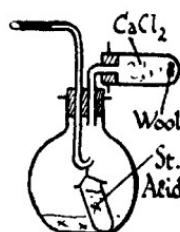


FIG. 156

4. *To determine the Mass of Carbon Dioxide evolved by the action of an Acid on a given mass of a carbonate :—*

This is done, as a rule, by "difference," i.e. the carbon dioxide is expelled into the air, and the residue is weighed ; which is very simple in principle, e.g. perform the following experiments :—

(a) Half fill a small flask (2 oz. capacity) with dilute hydrochloric acid, and accurately weigh it ( $= a$  grams).

(b) Accurately weigh a small lump of marble ( $= b$  grams).

(c) Drop the marble into the flask and allow the acid to dissolve it completely. Carbon dioxide is expelled.

(d) When action has ceased, weigh again ( $= c$  grams).

Then  $b$  grams = mass of carbonate taken,

and  $a + b - c$  = mass of carbon dioxide evolved.

$$\text{Percentage of carbon dioxide in marble} = \frac{100(a + b - c)}{b}$$

Compare the result obtained with the correct percentage (44 per cent.). The error is due to the following causes :—

(a) Some of the carbon dioxide is left dissolved in the liquid. This tends to decrease the percentage.

(b) Water is evolved with the gas. This tends to increase the result.

(c) Carbon dioxide fills the flask in place of air. This decreases the percentage.

The first error can be corrected by *just* boiling the liquid over a small naked flame after all action in the cold has ceased ; the third by sucking out the carbon dioxide ; the second necessitates a special device for drying the gas as it passes out.

Fig. 155 shows a simple method, using cotton wool ; the gas is sucked out with a glass tube, not shown in the figure. In Fig. 156 a drying-tube filled with pieces of fused calcium chloride, or pumice moistened with strong sulphuric acid, is used. The carbonate is weighed into the flask ; the small test tube contains strong hydrochloric acid.

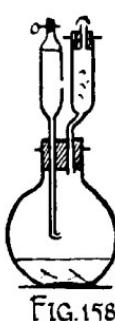
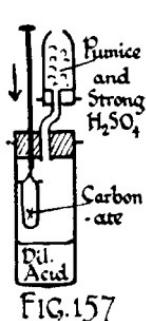


Fig. 157 is merely a modification of Fig. 156. *Dilute* acid is put into the tube ; the carbonate is weighed into the small test tube, which contains a small hole in the bottom.

Fig. 158 is similar in principle. The strong hydrochloric acid is contained in the pipette, and the entrance of it is regulated by a small clip.

Figs. 159 and 160 show other forms of drying apparatus in which a few drops of strong sulphuric acid are used in place of the moistened pumice.

Fig. 161 is Schrotter's, one of the standard pieces of apparatus for this determination. The carbonate is put in by removing the stopper. The pipette contains the acid to decompose it, and the drying arrangement contains strong sulphuric acid.

Fig. 162 is a very efficient apparatus designed by the author some years ago and made by Messrs. F. E. Becker & Co., of Hatton Wall, London.

To use the apparatus, remove the rubber stopper carrying the pipette and drying bulb G. Weigh the test tube, both before and after putting in the carbonate (difference in mass = weight of carbonate used). Into the small test tube C put a few drops of strong sulphuric acid. Remove stopper, and by suction, fill the pipette A with acid, say hydrochloric, pinch the rubber tube B, and replace. Put into E a little water, replace the stopper and hang the whole to the specific-gravity hook of a balance and weigh.

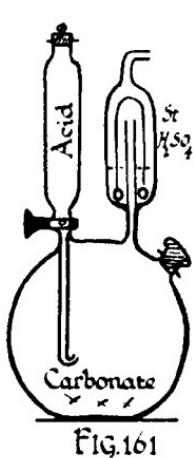


FIG. 161

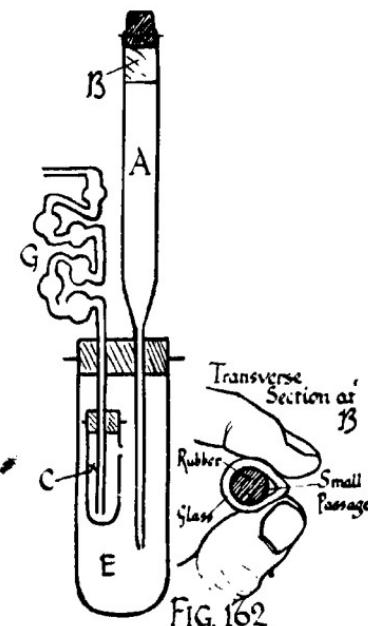


FIG. 162

Slightly squeeze the rubber tube B between the finger and thumb, as in diagram, and allow a few drops of acid to drop from A. The liberated gas escapes through the drying apparatus G. When the action is over, remove the stopper, and attach B to a suitable apparatus, and aspirate until the carbon dioxide in E is eliminated. Replace stopper and weigh the whole apparatus—the loss of weight represents the evolved carbon dioxide.

If still greater accuracy is desired, it is well to arrange this apparatus in combination with tubes and aspirator as shown in Fig. 163.

A is the decomposition apparatus used as described above.

It is connected at 1 with B, which will absorb the carbon dioxide evolved from A.

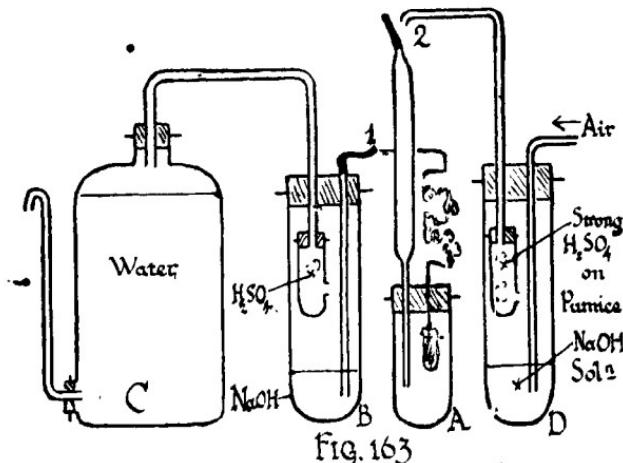
The gain in weight of B should equal the loss in A.

C is an aspirator by means of which a current of air can be drawn through A in order to expel every trace of carbon dioxide from it.

To do this, *after the decomposition of the carbonate is complete*, the top of the acid pipette is connected at 2 with the tube D, through which the air has to pass before it reaches A. D contains caustic soda and sulphuric acid to ensure that when it enters A it contains no moisture and carbon dioxide.

5. To determine the Mass of Carbon Dioxide evolved by the action of heat on a carbonate:—

*Not all* carbonates are decomposed by the action of heat. Use for this exercise precipitated chalk or magnesite.



Thoroughly dry a porcelain crucible by heating it for five minutes over the bunsen flame, cool, and weigh it very accurately. Put in sufficient of the carbonate to form a layer about one-eighth of an inch thick on the bottom of the crucible, and reweigh to find the amount used.

Heat strongly over the bunsen flame or put in a muffle furnace for one hour, with the lid of the crucible removed.

Cool in a desiccator and weigh again. Find and record the loss that has occurred. Reheat in a similar manner for ten or fifteen minutes, cool and reweigh. Find and record the second loss. If it exceeds 0.001 grams, repeat the heating until no greater loss is obtained, when it may be assumed that the carbonate is completely decomposed.

Find the *total loss* and calculate to a percentage. The weighings should be recorded as follows:—

Mass of crucible + Carbonate = gm.

“ “ only = ”

“

Then amount of carbonate used = gm.

Crucible + Carbonate before heating = ”

“ + “ after „ = ”

“

First loss = gm.

Crucible + Carbonate after first heating = ”

“ + “ second „ = ”

“

Second loss = gm.

and so on.

6. *Black's Researches on Chalk*, or to compare quantitatively the composition (with respect to the proportions of the two oxides they contain) of marble, Iceland spar, egg shell, precipitated chalk, oyster shell, etc.

(a) Determine the percentage of calcium oxide present in each of the following substances—express as  $\text{CaO} = x$  per cent.

(b) Determine the percentage of carbon dioxide present in the same substances—express as  $\text{CO}_2 = y$  per cent.

1. By action of heat alone. 2. By action of acid. Substances to be used: Precipitated chalk, marble, Iceland spar, egg shell (cleaned and dried), oyster shell, calcite, calc spar.

Methods to be followed are those just described. Take care to powder the substance thoroughly if it is not in that condition, and use small quantities for the experiments.

Collect and tabulate the results:—

Substance used.	Composition.		
	Percentage CaO.	Percentage $\text{CO}_2$ .	Percentage Total.
Chalk . . . .			
Marble . . . .			

From an inspection of the series of results, what deductions do you make ?

7. To determine the Volume of Carbon Dioxide evolved by the action of an acid on a carbonate.

Fit up the apparatus as shown in Fig. 164. Test to see if it is perfectly air-tight ; until it is so, it is useless to proceed.

Weigh out into the flask (preferably by use of a weighing-bottle) a few grams of the carbonate. Cover it with about 10 c.c. of water. Fill the pipette with strong hydrochloric acid, and refit the flask. Liberate the acid a few drops at a time until the carbonate has completely dissolved ; then warm the liquid to expel the dissolved gas. Allow the gas to cool down to the temperature of the laboratory,

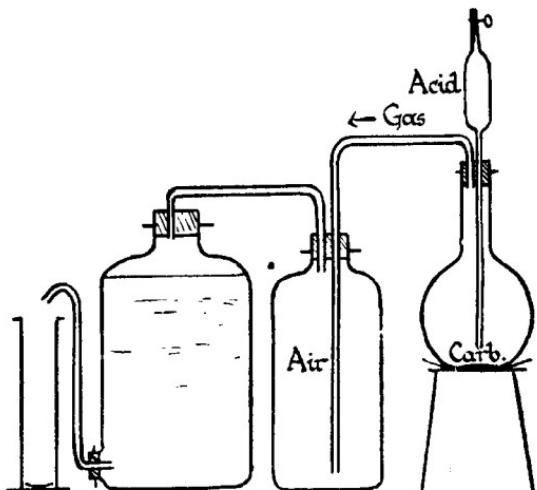


FIG. 164

taking care to keep the end of the delivery tube from the aspirator under the surface of water in, or from, the collecting vessel.

Measure the volume of water collected, deduct the volume of acid run into the flask, and if an accurate result be required, correct this volume to N.T.P. Calculate the volume of gas obtainable from 1 gram of the carbonate.

Why is the "Winchester" used ? Would it be necessary (say) in the case of hydrogen being the evolved gas ?

Why should the tube leading from the flask go to the bottom of the Winchester, and that which leads out of it come from the top ?

What relationship must there be between the volume of the Winchester and the volume of gas evolved ?

Perform the experiment at least twice and take the average of the results if they are nearly identical.

Carbon monoxide or carbonic oxide is the lower oxide of carbon. It is of considerable industrial importance, being used in metallurgical operations, reverberatory furnaces for reducing purposes, as an adulterant for coal gas, etc.

*Methods of Preparation :—*

1. Pass carbon dioxide over heated carbon (Fig. 165). An iron

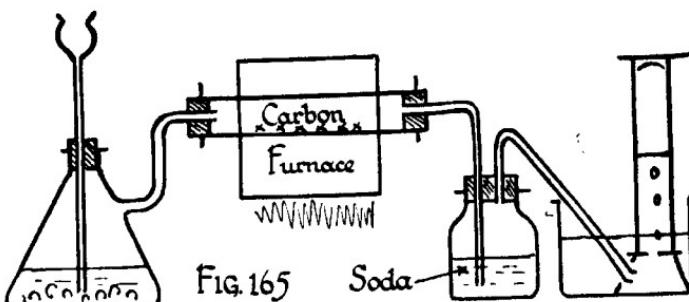


FIG. 165

Soda

tube should be used, and the stream of carbon dioxide should be a slow one.

The same preparation goes on in a clear fire (Fig. 166). The carbon dioxide first formed is reduced in the centre by the hot coke to carbon monoxide, which then burns to produce the dioxide again.

FIG.  
166

2. Pass air over strongly heated coke or anthracite. A mixture of carbon monoxide and nitrogen is obtained, known commercially as "generator" or "producer" gas.

3. Pass steam over red-hot anthracite or coke. A mixture is produced known as "water gas." This gas, which has an average composition of 50 per cent. hydrogen, 40 per cent. carbon monoxide, 5 per cent. carbon dioxide, and 5 per cent. nitrogen, is used largely as gaseous fuel and as a reducing agent. It can be made very luminous by mixing it with a very small proportion of unsaturated hydrocarbons, and in this form it is used in America in lieu of coal gas.

4. From formic acid, by heating it with strong sulphuric acid. The former can be looked upon as a compound made up of water ( $H_2O$ ) and carbon monoxide (CO). Hot sulphuric acid abstracts the water and liberates the gas.

5. The usual lecture preparation is from oxalic acid and strong sulphuric acid (Fig. 167).

Oxalic acid may be looked upon as being composed of water ( $H_2O$ ), carbon dioxide ( $CO_2$ ), and carbon monoxide ( $CO$ ). When heated with strong sulphuric acid, the water is abstracted and both oxides of carbon are liberated. The dioxide is scrubbed out by passing the mixture through two wash bottles containing baryta water, and then through two containing strong caustic soda solution.

The gas is present in coal gas in small quantity, in the vapour evolved from lime kilns, and in blast furnace gas. It is produced at all times when carbon is burnt in an insufficient supply of air—hence “slow combustion” stoves, such as are often met with on the Continent, are liable to emit it, and many fatal accidents have occurred due to defective ventilation in connexion with their use. Zola’s death was due to this cause.

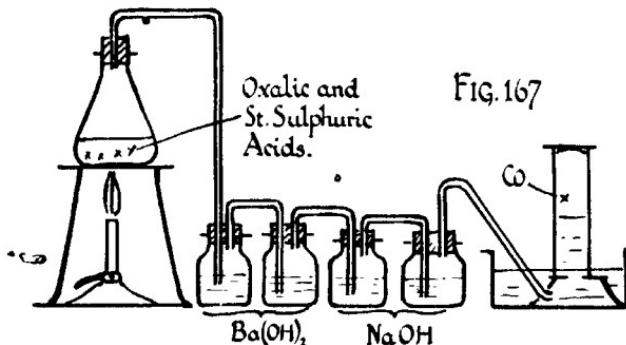


FIG. 167

#### PROPERTIES

It is a colourless gas, tasteless and practically odourless; very slightly soluble in water, and a little lighter than air. It is extremely poisonous—1 per cent. in air is fatal to human beings. It burns with a blue lambent flame, the temperature of which is  $1,400^{\circ}C.$ , to form carbon dioxide; it does not support combustion, and has no action on lime water or litmus paper.

It explodes when mixed with an equal volume of air or half its own volume of oxygen, and is extremely difficult to liquefy.

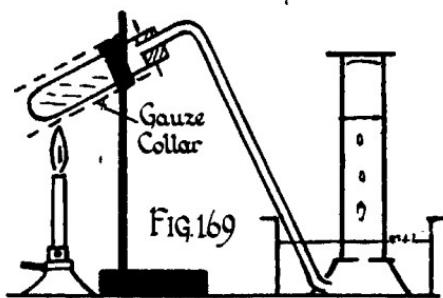
It acts as a powerful reducing agent at high temperatures. It is absorbed by a solution of cuprous chloride in hydrochloric acid (Fig. 168). The gas is delivered up the Crum tube and a solution of cuprous chloride passed from the cup a few drops at a time. As the gas is absorbed, the mercury rises.

It unites directly with certain elements, for example nickel, potas-

sium, and iron, to form carbonyls. The carbonyl of chlorine,  $\text{COCl}_2$ , is known as phosgene gas, which on exposure to moist air forms hydrochloric acid gas and carbon dioxide. It was used in the Great War as a "poison" gas.

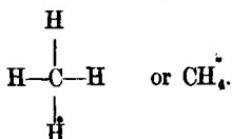
*Marsh gas or methane* is a gas which is produced by the decay of vegetable matter, particularly in swampy districts. A sample may be collected often from a stagnant pool by piercing the bottom mud with a stick. The bubbles of gas as they reach the surface may be ignited. A large percentage of coal gas is methane.

It is prepared usually for experimental purposes by heating a mixture of fused sodium acetate and soda lime in a test tube provided with a collar of wire gauze, to prevent cracking the glass (Fig. 169).

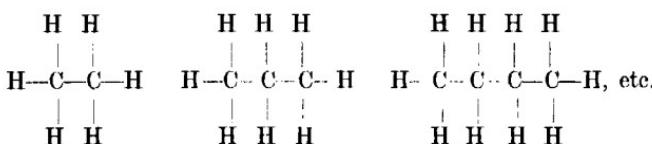


The gas produced will be found to be lighter than air, and to burn with a fairly luminous flame to form water vapour and carbon dioxide. It is also explosive when mixed with air or oxygen.

Analysis of the gas shows that it is composed of hydrogen and carbon only; it is therefore termed a *hydrocarbon*. The proportion by weight in which the constituents exist is 12 of carbon to 4 of hydrogen; the "picture" of the molecule is thus given as:—



By other chemical reactions it is possible to prepare or isolate from natural substances, like crude rock oil, other hydrocarbons, e.g. ethane, propane, butane, pentane, hexane, etc., and analyses of these compounds show that their molecular compositions are represented by the formulae  $C_2H_6$ ,  $C_3H_8$ ,  $C_4H_{10}$ ,  $C_5H_{12}$ ,  $C_6H_{14}$ , etc., the increases in molecular weights being identical ( $CH_2$ ). Such a series of compounds is known as an *homologous series*, and the graphic formulæ used to represent them are derivable from marsh gas by substitution of the  $CH_3$  or *methyl group* in place of the hydrogen atom at the end of the chain.



Theoretically, there will be an infinite number of possible hydrocarbons of this series ; as a matter of fact a very large number has been isolated from American rock oil, which is, to all intents and purposes, a mixture of them.

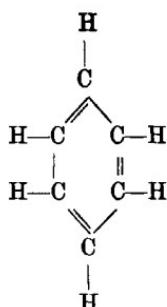
When the crude petroleum is fractionally distilled—that is, heated to a gradually increasing temperature, the distillates being carefully collected separately as they are evolved at the various temperatures—first gases, then liquids of low boiling points, and then liquids of higher boiling-points are obtained which are used on an enormous scale in everyday life, as petrol, paraffin, lubricating oils, salves, etc.

These fractions, known as *mineral oils*, are all mixtures of hydrocarbons of the paraffin series (as it is termed), the first containing the lower members of the series, and the latter the higher ones.

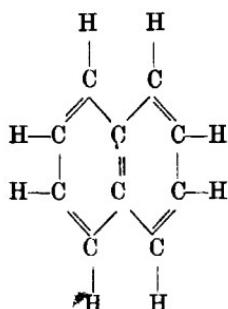
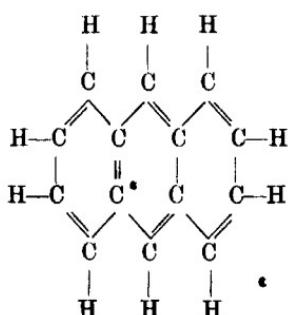
They are very stable and inert substances, resisting the action of most chemical reagents, particularly acids and alkalis ; consequently a mineral oil stain is very difficult to remove from a fabric.

In Scotland (and, it is now claimed, in some parts of England) certain shale deposits when distilled in a similar manner yield similar oils, the residual portion of which is known as *paraffin wax*.

The great Russian deposits contain benzoline or benzine, a mixture of higher members of the same series. Coal tar yields benzene ( $C_6H_6$ ), a hydrocarbon which can not be correctly represented as an open chain. Kekulé suggested that carbon and hydrogen were arranged as a “ring” :—

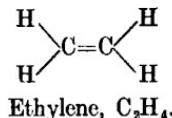
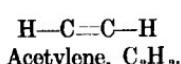
Benzene,  $C_6H_6$ .

Benzene and other hydrocarbons of the same series are obtained from the destructive distillation of coal tar, e.g. naphthalene and anthracene:—

Naphthalene,  $C_{10}H_8$ .Anthracene,  $C_{16}H_{10}$ .

Naphthalene is used as the starting-point in the synthetic preparation of indigo in one important process, and anthracene is the raw product from which is manufactured alizarine (the active colouring principle in madder), used in Turkey red dyeing.

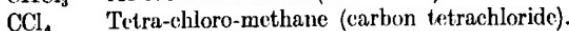
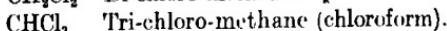
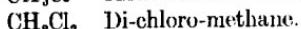
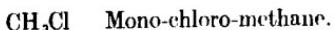
Other hydrocarbons are known, e.g. acetylene and ethylene, which are considered to be "unsaturated" compounds.



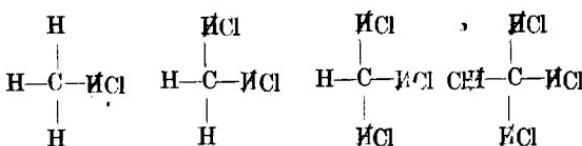
### Substitution Compounds

"When methane is mixed with chlorine and exposed to sunlight, a violent reaction occurs, but when the chlorine is diluted with carbon dioxide, and allowed to act gradually, chlorine substitution products are obtained."

An analysis of these compounds shows that their molecular composition is represented by the formulae :—



It is evident that these compounds are methane in which the hydrogen has been replaced by chlorine ; in other words, a substitution has taken place :—

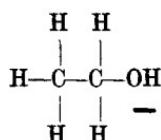
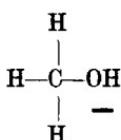


*Chloroform* is not yet prepared by this process commercially, the usual way being to act on bleaching powder with ethyl alcohol and distil the mixture on a water bath (Fig. 170).

Suitable quantities to use are 180 grams of bleaching powder, 400 c.c. of water, and 11 c.c. of alcohol in a one-litre flask. The heating must be gradual, and there is considerable frothing. The distillate contains alcohol and water as well as chloroform, which settles to the bottom of the mixture.

The sp. gr. of chloroform is 1.5, and its boiling-point is  $61.5^{\circ}\text{C}$ .

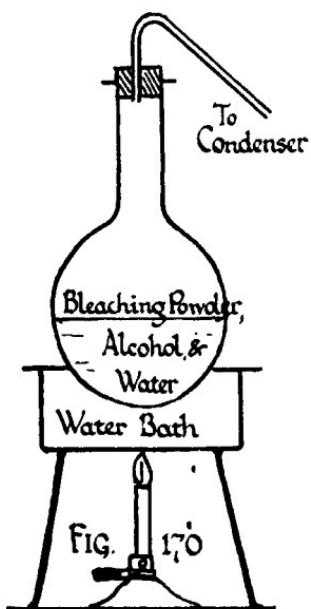
**Alcohols.** This class of compounds can be looked upon as hydroxyl (O—H radicle) substitution products of the hydrocarbons, e.g.:—



Methyl alcohol,  $\text{CH}_3\text{OH}$ .

Ethyl alcohol,  $\text{C}_2\text{H}_5\text{OH}$ .

Again we find an homologous series. More than forty alcohols were prepared by Dr. R. H. Pickard, F.R.S., and his research assistants in the chemical laboratories of the Blackburn Technical College a few years ago.



*Methyl alcohol*, also known as wood spirit, and wood naphtha, is obtained in the destructive distillation of wood and beetroot sugar refuse. Its chief uses are as a solvent for gums and resins in the varnish industry; in the preparation of coal-tar dyes; and as an adulterant of ethyl alcohol, in the preparation sold as methylated spirit.

*Ethyl alcohol*, or ordinary alcohol, is prepared by the fermentation of sugar, the weak solution thereby produced being fractionally distilled.

Boiling-points      Methyl =  $66^\circ\text{ C.}$

Ethyl =  $78.3^\circ\text{ C.}$

Specific gravity (at ordinary temperatures)      Methyl = 0.793

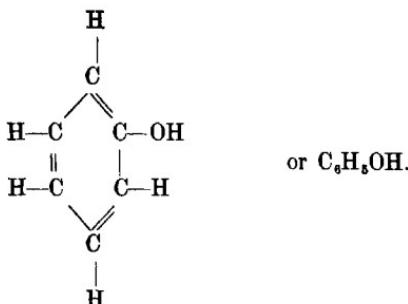
Ethyl = 0.8

*Amyl alcohol* or fusel oil is a mixture of two or more alcohols higher in the series than ethyl alcohol.

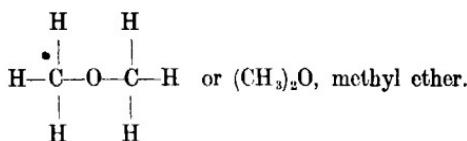
*Glycerol* (glycerine) is also an alcohol, and contains three hydroxyl groups— $\text{C}_3\text{H}_5(\text{OH})_3$ .

*Cetyl alcohol*,  $\text{C}_{16}\text{H}_{32}\text{OH}$ , is present in spermaceti wax. Chinese wax and beeswax contain alcohols higher still in the fatty series.

*Phenol* (commonly known as carbolic acid) is a compound of the benzene series which resembles an alcohol in some respects:—



**Ethers.** This class of organic compounds is analogous to oxides, the basic radicle being replaced by one or more hydrocarbon radicles, e.g.—



$(\text{C}_2\text{H}_5)_2\text{O}$ , or ethyl ether, i.e. ordinary or sulphuric ether.

$\text{CH}_3\text{O.C}_2\text{H}_5$ , or methyl ethyl ether.

The two latter may be prepared by what is known as the continuous etherification process. Ethyl alcohol or methylated spirit is carefully run into half its own volume of strong sulphuric acid, without allowing the temperature to rise unduly. It is then distilled at a temperature of  $140^\circ \text{ C.}$  (in the liquid), a slow stream of alcohol or methylated spirit being passed in as the ether distils off (Fig. 171).

Ether gives off dangerously inflammable vapour, and great care should be taken to keep flames away from it. *Bottles of ether should be kept on the floor and never on a laboratory bench. Very disastrous consequences have occurred due to inattention to this simple precaution.*

Ethyl ether, sp. gr. at  $15^\circ \text{ C.} = 0.7$ ; b.p. =  $35^\circ \text{ C.}$  Ethyl ether dissolves in

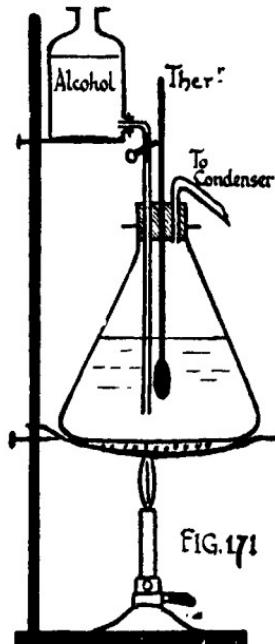


FIG. 171

ten times its own volume of water, and is miscible with alcohol and other organic liquids in all proportions.

*Chief Uses.* As a solvent for fats, oils, resins, etc., and as an anaesthetic in surgery.

**Aldehydes.** When a slow stream of air is passed through methyl alcohol some of it vaporizes; and if the gaseous mixture be passed over heated copper gauze, and then the product so formed into water, it will be found that a solution is obtained which has very characteristic properties (Fig. 172). It has a pungent smell, will reduce an ammoniacal solution of silver nitrate (oxide) to metallic silver, and will restore the colour of Schiff's reagent, which is made by decolorizing a dilute solution of magenta in water, with a stream of sulphur dioxide.

This solution is known as *formalin*, and it is possible to make it of 40 per cent. strength, in which condition it is usually sold.

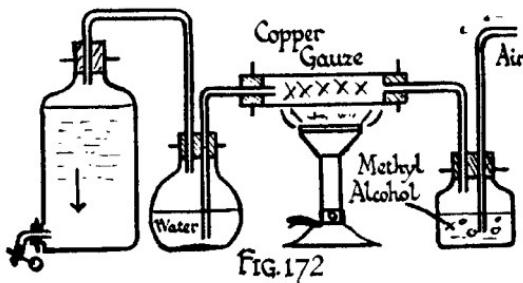
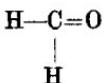


FIG. 172

It is used in large quantities in the manufacture of artificial dyes, and as an antiseptic.

The gas is termed *formaldehyde*, and it has been found to have the composition 2 parts by weight of hydrogen, 12 of carbon, and 16 of oxygen; in other words there are 2 atoms of hydrogen, 1 atom of carbon, and 1 of oxygen in the molecule, (or graphically)



Other compounds exhibiting similar properties and containing the radicle CHO have been prepared; and as they apparently are alcohols which have been deprived of two atoms of hydrogen, they are named aldehydes (*Alcohol dehydrogenatum*). The name Form indicates that on further oxidation the acid so obtained is formic.

The next member of the series is Acetaldehyde. Benzaldehyde, or artificial oil of almonds, or (in the crude form) oil of mirbane, is the simplest benzene aldehyde,  $\text{C}_6\text{H}_5\text{CHO}$ .

**Acids.** Hundreds of carbon acids have been prepared and their compositions investigated, and as a result it is firmly established that they all contain the monovalent group COOH, termed the carboxyl radicle. The lowest member of the paraffin series is *formic acid*, H.COOH, the next is *acetic acid*, CH<sub>3</sub>.COOH.

When these acids react with alkalis to produce salts, it is the hydrogen in the COOH group which is replaced by the metallic base, e.g.:—

Sodium formate	.	.	H.COONa.
Potassium acetate	.	.	CH <sub>3</sub> .COOK.
Calcium acetate	.	.	(CH <sub>3</sub> .COO) <sub>2</sub> .Ca.

*Oxalic acid* is termed a *di-basic acid* because it contains two carboxyl groups. Its formula is written (COOH)<sub>2</sub>.

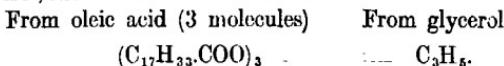
Sodium oxalate is therefore (COO)<sub>2</sub>Na<sub>2</sub>.

Calcium oxalate, (COO)<sub>2</sub>Ca.

Other important acids are:—

Butyric acid	C <sub>4</sub> H <sub>7</sub> .COOH	(in butter fat).
Palmitic acid	C <sub>15</sub> H <sub>31</sub> .COOH	(from palm oil).
Oleic acid	C <sub>17</sub> H <sub>33</sub> .COOH	(from olive oil).
Stearic acid	C <sub>17</sub> H <sub>35</sub> .COOH	(from tallow).

*Note.*—Fats are salts of the “fatty acids,” in which the hydrogen of the carboxyl group has been replaced by the tri-hydric alcohol glycerol, instead of a metal. Thus:—



For the preparation of these fatty acids, the fat is first *saponified* by boiling it with caustic soda or potash. Double decomposition occurs, the alkali salt of the acid being formed (which is known as a soap), together with glycerol.

The soap can then be hydrolyzed by the addition of a mineral acid, another double decomposition resulting with the formation of the fatty acid (often insoluble in water) and the production of the alkali salt of the mineral acid.

As fats always contain more than one glyceride, although one is usually in excess, further manipulations are necessary if a pure fatty acid is required.

**Acetic acid** is prepared by three processes:—

(a) Fermentation of dilute alcoholic solutions containing small amounts of nitrogenous and phosphatic food-stuffs, such as beer or light wines, by the microscopically small plant named *Mycoderma aceti* or mother of vinegar.

(b) By the quick vinegar process. A large vat (Fig. 173) is made into three compartments by means of two grids, the space between being filled with beech shavings. Holes are bored at intervals in the sides of the barrel to allow air to enter.

In the top compartment A vinegar is put, which slowly drops into B by travelling down the pieces of string that hang through the holes in the top grid. The shavings thus become covered with *Mycoderma aceti* present in the vinegar.

Dilute alcohol is now put into A, and it ultimately finds its way into C, from which it siphons off at intervals. After two or three passages the alcohol is completely oxidized to acetic acid.

(c) From pyrolignous acid, obtained by the destructive distillation of wood. Soda ash is added to the crude distillate to produce sodium acetate, which is crystallized out, purified by recrystallization, and then fused.

The fused sodium acetate is then treated with strong sulphuric acid and distilled, when acetic acid is evolved.

This is further purified by freezing out the acetic acid.

Anhydrous acetic acid has a m.p. of  $16.5^{\circ}\text{C}$ , and a b.p. of  $118^{\circ}\text{C}$ .

**Oxalic acid** may be prepared by oxidizing cane sugar (1 part) with strong nitric acid (6 parts); and a modification of this process is used industrially for

nearly the whole production of oxalic acid.

Sawdust is made into a paste with a strong solution of a mixture of equal parts of caustic soda and caustic potash, heated in iron pans to  $210^{\circ}\text{C}$ ; the mixed oxalates of sodium and potassium are extracted with water, and boiled with lime. The resulting calcium oxalate is decomposed with dilute sulphuric acid, separated from the calcium sulphate, concentrated, and crystallized.

**Formic acid** is prepared by heating a mixture of oxalic acid and glycerine and condensing the distillate; a temperature of  $110^{\circ}\text{C}$ . should be maintained whilst the process is in operation.

Formic acid is being used very considerably in the textile industry to-day, and with great success.

**Carbohydrates** (so called because the ratio of hydrogen to oxygen found in them is the same as that in water) form a class of compounds of complicated chemical structure, the nature of which is not yet thoroughly known.

There are two groups:—.

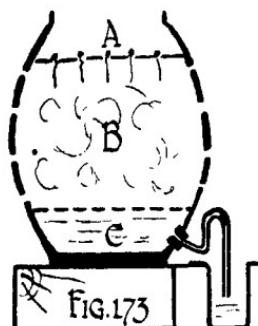


FIG. 173

1. *Sugars*: (a) The glucoses ( $C_6H_{12}O_6$ )<sub>n</sub>.

(b) The sucroses or cane sugar ( $C_{12}H_{22}O_{11}$ ).

2. *Starches and cellulose* ( $C_6H_{10}O_5$ ).

Glucose, grape sugar, or dextrose,  $C_6H_{12}O_6$ , occurs naturally in fruits and honey, and is also prepared industrially in large quantities from starch. One method in use is to raise to boiling-point water containing 1.5 per cent. sulphuric acid.

Into this is then run gradually a mixture of starch and water, and boiling is continued for half an hour. The mixture is neutralized with chalk, and concentrated, the calcium sulphate being precipitated. The clear syrup is further concentrated in vacuum pans until it is a thick viscous liquid or a solid.

It contains about 70 per cent. glucose, about 30 per cent. maltose, dextrose, and calcium salts of organic acids.

If the presence of a small quantity of salt be not objected to, hydrochloric acid may be used instead of sulphuric, and the neutralization effected with sodium carbonate ; the boiling should be continued for at least an hour.

A much purer substance can be obtained by boiling cane sugar with dilute acid (see invert sugar).

Glucose is less sweet than cane sugar. Its solubility is : 10 in 12 of cold water ; 1 in 50 of cold alcohol ; 1 in 5 of boiling alcohol. It is not blackened easily when treated with strong sulphuric acid, and it is immediately fermented by yeast.

It is used for making alcohol, in confectionery and jam, and by dyers and calico-printers as a thickening ingredient.

*Cane sugar*,  $C_{12}H_{22}O_{11}$ , is the most important of the naturally occurring sugars, and forms an essential article of human diet. Many vegetables and plants contain it, some in sufficient quantities to pay to extract it, e.g. sugar cane, maple tree, beetroot.

It is very soluble in water, dissolving to the extent of 67 in 23 at 20° C., and to almost any degree in hot. It is insoluble in alcohol, fuses at 160° C., and does not crystallize on cooling. It is readily blackened by strong sulphuric acid, and is very sweet. It is not fermentable by yeast until changed to glucose, and does not reduce Fehling solution.

*Levulose* is found in many ripe fresh fruits and in honey ; it is sometimes known as fruit sugar. It is much sweeter than glucose and nearly as sweet as cane sugar.

*Honey* is a mixture of one-half levulose, one-third to one-half dextrose, and some cane sugar. The former is soluble in cold alcohol, and dextrose in boiling alcohol.

*Lactose or milk sugar*,  $C_{12}H_{22}O_{11} \cdot H_2O$ , is present in milk, and can be obtained by concentrating and crystallizing the whey. \*Cow's milk

contains 4·7 per cent. When heated to 130° C. the water of crystallization is evolved. It is sweeter than cane sugar, but less soluble, e.g. 1 in 6 of cold water, 1 in 2·5 of hot water, and insoluble in alcohol.

*Maltose*,  $C_{12}H_{22}O_{11} \cdot H_2O$ , is formed from starch by the action of a ferment, diastase, produced when barley germinates. Grain containing this ferment is known as malt. The best temperature for the conversion of starch into sugar by malt is 65° C. Its chief use is as a fermentable sugar in the preparation of alcohol, as it very readily ferments with yeast. It is less soluble in alcohol than dextrose.

*Suitable formula for its preparation* : Into 350 c.c. of boiling water run 100 grams of starch completely mixed with 100 c.c. of cold water, and stir well. When the temperature has fallen to 65° C., add 7 grams of crushed malt, and keep the mixture at 65° (on a water bath) for one hour. At the end of that time test for sugar and starch. When maltose is boiled with dilute acids it is converted into glucose.

*Invert sugar* contains equal quantities of dextrose and levulose. It can be prepared by dissolving cane sugar in water, adding a little sulphuric or hydrochloric acid, and keeping on a water bath for half an hour at 100° C.

The acid is neutralized by addition of barium carbonate or caustic soda, and evaporated to a syrup. A great deal of artificial honey is prepared in this manner.

*Starch*,  $(C_6H_{10}O_5)_n$ , is a carbohydrate of universal distribution in vegetable tissues (see pages 184–188). It is insoluble in most solvents in the cold, but when boiled with water and solutions of certain chlorides, etc., it forms colloidal solutions which on cooling set as “jellies.”

Boiled with dilute acids it is converted into sugar. Heated with strong sulphuric acid it is “carbonized.” It is decomposed in a similar manner when heated alone in the dry condition.

The reaction for the identification of starch is that of iodine, which forms a compound with it that gives a deep blue colour on dilution with water.

## SECTION XI

### CHLORINE

**C**HLORINE is a greenish gas which is very readily obtained from hydrochloric acid. Scheele, who first prepared it in 1774 from this acid and manganese dioxide, named it dephlogisticated marine acid air, a name which was changed some forty years later by Davy to chlorine, on account of its greenish-yellow colour. It is an element. •

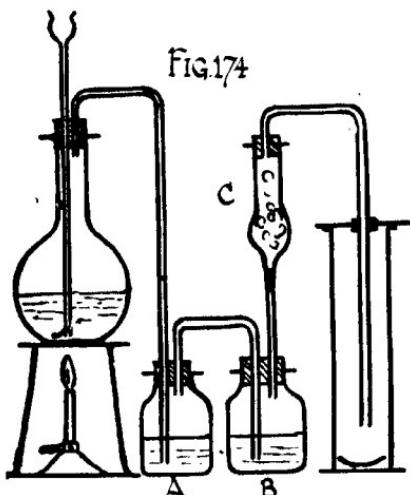


FIG.174

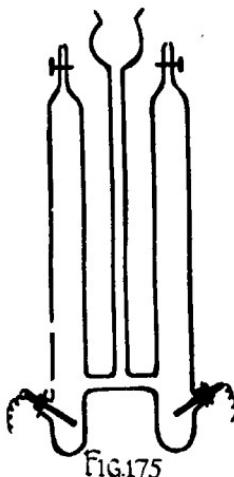
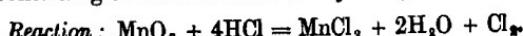


FIG.175

It is still prepared chiefly by the original method, although many peroxides heated with hydrochloric acid will evolve the gas.

A convenient apparatus for its preparation on a small scale is shown in Fig. 174. A and B are wash bottles containing water; C is a tube containing calcium chloride to dry the gas.



It may also be obtained from many chlorides by heating with

strong sulphuric acid and a peroxide. With common salt the reaction is :—



When a strong solution of hydrochloric acid is electrolyzed, chlorine is evolved at one electrode (Fig. 175).

It can also be obtained by heating hydrochloric acid with either potassium dichromate, potassium chlorate, red lead, or bleaching powder.

Before collection, which may be by displacement of air or over strong brine, the gas should always be washed through water to collect any hydrochloric acid that may be carried over.

#### SOME CHEMICAL PROPERTIES

1. A jet of hydrogen or coal gas burns in a jar of chlorine to produce hydrochloric acid (Fig. 176).

2. A solution of chlorine in water exposed to sunlight gradually loses its colour, liberates oxygen, and forms a solution of hydrochloric acid (Fig. 177).

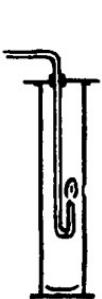


FIG.176

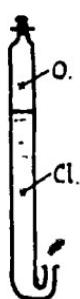


FIG.177

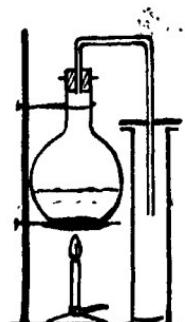


FIG.178

3. Mix chlorine water and sulphuretted hydrogen solution : sulphur is deposited and hydrochloric acid is produced.

4. When phosphorus (on a deflagrating spoon) is placed in a jar of the gas it melts, spontaneously inflames, and produces fumes of phosphorus chloride.

5. Burning sodium in it produces a white deposit of common salt.

6. It bleaches moistened vegetable-coloured articles, due to liberation from the water of "nascent" oxygen, which oxidizes the colouring matter.



*Note.*—Perfectly dry chlorine will not bleach.

7. It attacks mercurv.

8. It unites with hydrogen in daylight to form hydrochloric acid.
9. Finely powdered metals like antimony, iron, copper, burn when dropped into chlorine, particularly if they are warm.
10. It combines with hydrogen present in hydrocarbons, such as turpentine, to form hydrochloric acid. The energy of combustion is usually sufficient to inflame the rest of the turpentine.
11. Note effect of the gas on a solution of potassium iodide. Iodine is liberated, which in the presence of starch produces a blue compound.

#### PHYSICAL PROPERTIES

Irritating odour; green colour; 1 litre at N.T.P. weighs 3.1645 grams;  $2\frac{1}{2}$  times as heavy as air; fairly easily liquefied; soluble in water to a moderate degree.

*Chief Uses.* As a bleaching agent for cotton goods; a disinfectant and deodorizer; for preparation of chlorides and other compounds containing chlorine; for extraction of gold from quartz.

#### LABORATORY EXERCISES

Fit up the apparatus as shown in the diagram (Fig. 178). In the flask put some commercial hydrochloric acid and manganese dioxide. Thoroughly mix and then gently warm.

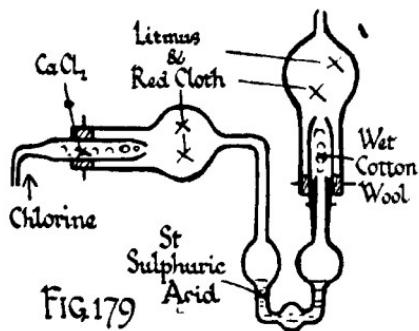


FIG. 179

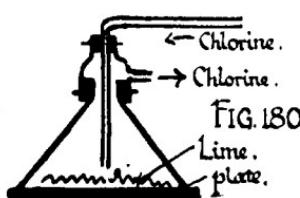


FIG. 180

Collect two or three samples of the gas in *dry* gas jars.

Is the gas heavier or lighter than air? Is it combustible? Will it support combustion (a) of an ordinary light? (b) of turpentine on filter paper? (c) of phosphorus (not previously ignited)? (d) of sodium (ignited first)?

Drop in a few grains of powdered antimony and describe what happens.

Test its action—wet and dry—on litmus paper and Turkey red cloth (Fig. 179).

Prepare a solution of the gas in water, describe its colour and smell.

Pass the gas into a few c.c. of a fairly strong solution of caustic soda. Note what happens when dilute acid is added to the product formed.

Pass the gas through slaked lime, using the apparatus shown in Fig. 180. Study the effect of adding an acid to the substance produced.

*On the commercial scale* at the present day chlorine is prepared :  
(1) By the original Scheele method ; (2) by the Deacon process ;  
(3) by electrolytic methods ;—of which the first is the most important.

In the first method the reaction vessel is made of stone slabs, luted and bound together by iron bands, and is known as a chlorine still (Fig. 181).

Manganese dioxide is spread on a raised wooden floor, the acid being fed in through a funnel which dips into a bowl placed on this floor, over which it gradually flows.

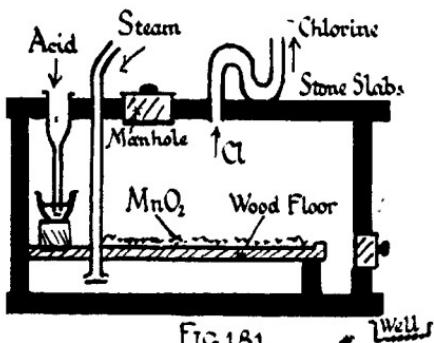


FIG. 181

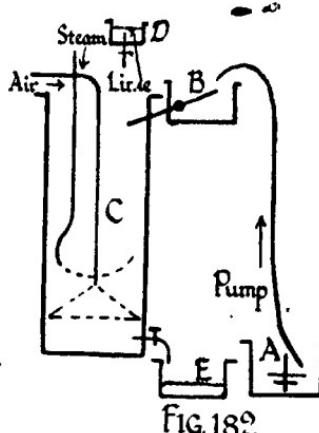


FIG. 182

Here manganic chloride is formed as a dark brown liquid. Steam is then passed in, under the wooden floor of the still, with the result that the higher chloride of manganese is decomposed to form the lower manganous chloride, with liberation of chlorine.

This process would not be a commercial success unless the manganese could be "recovered." This is done by the Weldon process. Fig. 182 shows the principle of this method.

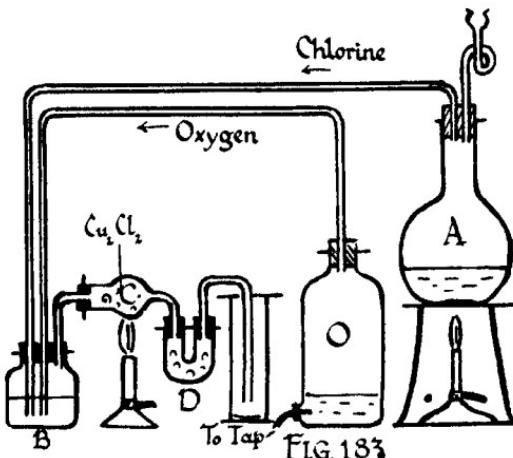
The still liquor is neutralized with chalk in A. It is then pumped to the settling tank B, from which the supernatant liquid (which contains manganous chloride and calcium chloride) is run at intervals into the oxidizer C.

Here it is mixed with milk of lime from D, the temperature raised to  $50^{\circ} C$ . by blowing in steam, and finally air is blown through. This produces an insoluble compound of calcium and manganese,  $CaOMnO_4$ .

or  $\text{CaO}_2\text{MnO}_4$ , which can be again treated with hydrochloric acid to yield chlorine.

For this purpose the contents of C are run into settling tanks E, where the precipitate settles as a paste known as Weldon mud. After the supernatant liquid has been decanted off the residue is passed into the chlorine stills.

The Deacon process can be illustrated by using the apparatus shown in Fig. 183. Hydrochloric acid gas is generated in flask A from salt and sulphuric acid, oxygen is passed from aspirator O, and both gases are dried by passage through strong sulphuric acid in bottle B. They then pass through the hard glass tube C, containing heated cuprous chloride, in contact with which the dry hydrochloric acid is decomposed, hydrogen combining with oxygen, and chlorine being liberated. By passing the product through D, the water can be absorbed and dry chlorine collected in the gas jar.



Owing to technical difficulties in working the process, the method has not developed to the extent that was once thought possible.

One of the most successful methods for the electrolytic preparation of chlorine is that of Castner (Fig. 184).

The reaction chamber is divided into three cells by two vertical partitions which go nearly to the bottom, along which is spread a thin layer of mercury. The two outside compartments, which are duplicates, contain carbon electrodes, and the centre compartment an iron one. When the current is passed, the solution of sodium chloride in the outside compartments is decomposed with liberation of chlorine (which leaves the chamber by means of the pipes) and sodium which

dissolves in the mercury. The sodium amalgam thus formed is now sent into the centre compartment by rocking the cell, where it is decomposed by the water therein, thus producing a solution of caustic soda with liberation of hydrogen. As soon as the apparatus is rocked in the opposite direction the mercury passes back into the outside compartment to be used over again. The rocking action is made automatic by arranging one end of the cell on a pivot, and the other on an eccentric, with the motion of which the end resting on it gradually rises and falls.

**Hypochlorites.** If a stream of chlorine gas be passed into a solution of cold caustic soda or caustic potash, the gas is absorbed; and if the product be treated with very dilute acid and distilled under reduced pressure and at a low temperature, a solution is obtained which contains a very unstable acid called *hypochlorous acid*, the composition of which is represented by the formula  $\text{HClO}$ .

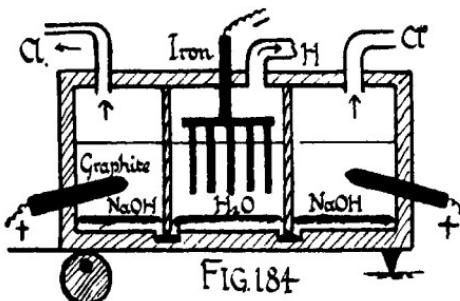


FIG. 184

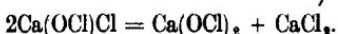
It is a salt of this acid which is produced, together with the alkali chloride, when chlorine is absorbed by cold caustic soda or potash.



This substance was prepared as long ago as 1785 by Berthollet, and called Eau de Javelle, from the name of the suburb of Paris where it was made in 1792. When Labaraque discovered in 1820 that a similar reaction occurred with caustic soda, the liquid was made from it instead of the more expensive potash, and sold under the name of Eau de Labaraque.

The most striking property of these hypochlorites, even in very dilute solution, is their power of bleaching—which is much more pronounced than with chlorine alone; this has been attributed to the liberation of hypochlorous acid, which is a bleaching agent, due to the fact that it very easily splits up into hydrochloric acid and nascent oxygen.

In 1798 a process was patented by Tennant for using lime instead of potash, the patent rights of which were subsequently revoked because it was proved that this "bleaching powder" had been previously in use in Lancashire. This substance is not calcium hypochlorite : what it really is has been the subject of much discussion. The most generally accepted view is that if pure it is a compound intermediate between calcium hypochlorite and calcium chloride, named calcium chloro-hypochlorite, the composition of which is given by the formula  $\text{Ca}(\text{OCl})\text{Cl}$ . This on treatment with water yields both calcium chloride and calcium hypochlorite.



*Bleaching powder*, sometimes incorrectly called chloride of lime, is made in large chambers, the floors of which are covered with a layer of fresh-slacked lime about four to six inches deep, raked into furrows to expose a greater surface. Chlorine straight from the chlorine stills is passed in until the gas is no longer absorbed. The excess is removed by blowing in a little powdered lime, and it is then collected and packed in barrels.

*Sodium hypochlorite*, for which there is now a considerable demand, is prepared electrolytically as well as by direct absorption of chlorine by caustic soda.

The liquid used is a solution of salt in water, the strength varying with the strength of the current. The electrodes are of carbon, and a constant circulation is kept up in the cell, so that the chlorine liberated at one electrode shall be absorbed by the caustic soda formed at the other. This process is described in greater detail under "Bleaching" (Section XVI, pages 205-213).

An emulsion of bleaching powder mixed with a solution of sodium carbonate also produces sodium hypochlorite and calcium carbonate. The latter may be sedimented or filtered off, and a solution of the former obtained.

Made by the last process the liquid contains dissolved lime. When prepared by means of the electric current it is, or should be, neutral, and when prepared in the original way it is strongly alkaline, due to the presence of free soda.

Besides being used for bleaching purposes under various trade names such as Parozone, Lavozone, etc., it is also used as a germicide (in which capacity it is highly efficient) as Chloros, Milton, etc., and as a "softening" preparation for size, under the name of Paetchner's solution.

The most suitable laboratory method for preparation of the liquid is to pass chlorine through 20 per cent. caustic soda solution till no more gas is absorbed, using the apparatus shown in Fig. 185.

**Sulphur dioxide ( $\text{SO}_2$ ).** Several oxides of sulphur are known, of which the dioxide is by far the most important.

It is obtained by burning sulphur in air or oxygen. Besides this method there are other ways by which the gas may be prepared:—

1. The usual laboratory method. Act on certain "heavy" metals with *strong hot sulphuric acid*. In this case the oxide is liberated from the acid, and a metallic sulphate and water are formed. The metals which will give this reaction are copper, mercury, silver. Copper, being the cheapest, is used.

To prepare the gas, set up the apparatus shown in Fig. 186.

2. *From sulphides* by roasting in a current of air or oxygen, e.g.

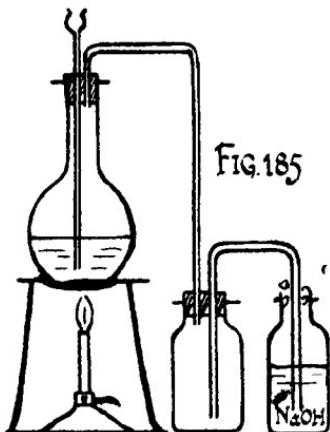


FIG. 185

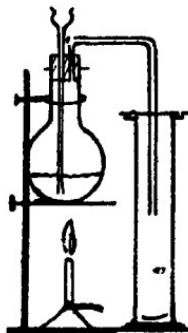


Fig. 186

iron pyrites, copper pyrites, cinnabar. The metal is converted in part or entirely into the oxide in most cases. This is a very general metallurgical method adopted for freeing ores from a quantity of their sulphur.

3. By heating sulphur with certain peroxides, e.g. manganese dioxide, or with sulphuric acid, the acid being thereby reduced.

4. By decomposing the class of salts known as *sulphites*, e.g. sodium sulphite, or sodium bisulphite, with dilute acids.

#### PROPERTIES

**Colourless.** (The so-called white fumes of sulphur dioxide are due to traces of sulphuric acid and sulphur trioxide that are sometimes formed.) Suffocating smell (minute traces of the gas breathed for a short time appear to be beneficial rather than harmful). It is non-combustible, and will not support ordinary combustion, but if sodium

peroxide be dropped into a jar of the gas a few grains at a time, brilliant combustion results.

It is somewhat heavy (more than twice as heavy as air), and soluble in water to form an acid solution known as sulphurous acid.

1 volume of water at 0° C. dissolves 80 volumes of sulphur dioxide.

1	"	20° C.	"	39	"	"	"
1	"	40° C.	"	19	"	"	"

Boiling expels all the gas.

It is easily liquefied by pressure (and so comes into commerce in glass siphons and iron bottles), and by cooling (Fig. 187).

Pass the previously dried gas into a small bottle surrounded with a freezing mixture of salt, ice, and calcium chloride.

*Liquid sulphur dioxide* is a fairly mobile liquid : poured into water it freezes it, due to rapid evaporation. At 0° C. a pressure of 1½ atmospheres condenses the gas to a liquid. At ordinary pressures a tem-

perature of - 10° C. condenses it. It dissolves phosphorus, iodine, resins, etc.

*Uses.* Sulphur dioxide is largely used as a bleaching agent for goods which must not be bleached by chlorine, e.g. straw, silk, sponge, flannel, blankets, and wool articles generally.

Its action in bleaching is due to the gas decomposing the water, which must be present, to form sulphur trioxide, thereby liberating hydrogen, which reduces the colouring to a more or less colourless compound.

Alkalies often restore the colour, e.g. flannel which has been well washed with soap returns to its original yellow colour.

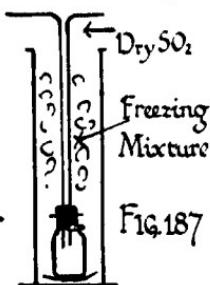
The gas is used for fumigating purposes, and in the liquid state it finds application as a refrigerating agent.

The best test for the identification of sulphur dioxide is its action on potassium chromate paper or solution, which it turns green.

*Hydrogen peroxide*, H<sub>2</sub>O<sub>2</sub>. Only two compounds of hydrogen and oxygen are known—water and hydrogen peroxide, which may be looked upon as oxide of water.

It is prepared by the action of several acids on barium peroxide, e.g. carbonic, hydrochloric, sulphuric, phosphoric, and hydrofluoric. In all cases the temperature must be kept low enough to prevent the decomposition of the hydrogen peroxide formed. If made with sulphuric acid, the acid must be diluted considerably.

On the commercial scale it is often prepared with the aid of phosphoric acid, but for laboratory purposes the best results are obtained by using hydrofluoric acid.



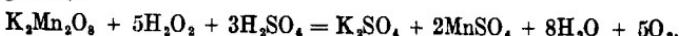
A well-made cigar box should be soaked in hot paraffin wax, and then coated with a layer of that substance; a suitable wooden stirrer should be treated in the same way.

The box should be partly filled with ordinary hydrofluoric acid diluted with four or five times its own volume of water. Barium peroxide is fed in with constant stirring until the liquid is no longer acid. The precipitate of barium fluoride is allowed to settle, when a strong solution of hydrogen peroxide will be found in the supernatant liquid.

Sodium peroxide may also be used, the reaction with hydrochloric acid being—



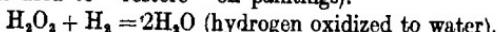
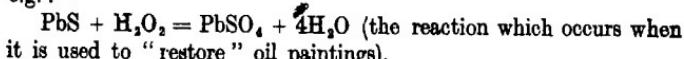
The aqueous solution obtained as above is concentrated *in vacuo*, over strong sulphuric acid. It comes into commerce labelled 10 volumes, 20 volumes, etc., which means that when treated with acidified potassium permanganate, 1 volume of solution yields 10 or 20 volumes of oxygen. (N.B.—Half of this, however, comes from the permanganate) :—



#### PROPERTIES

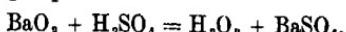
In the pure condition it is a colourless, odourless, syrupy liquid, of very bitter taste, sp. gr. 1·45, and very unstable.

Diluted with water, in which condition it is usually met with, it is a very active oxidizing substance, and a powerful bleaching agent, e.g. :—



#### LABORATORY EXERCISES IN THE PREPARATION AND ESTIMATION OF HYDROGEN PEROXIDE

1. *Preparation.* Weigh out about 30 grams of barium peroxide. To 40 c.c. of "bench" dilute sulphuric acid add 160 c.c. of water. Stir into this the barium peroxide, a gram or so at a time, keeping the temperature from rising. When all has been added, allow it to settle and pour off the clear supernatant liquid, which should be a dilute solution of hydrogen peroxide.



2. *Tests.* Add some of it to a solution of potassium iodide—iodine is liberated, and turns the solution yellow. Collect it by shaking up with two or three drops of chloroform—a violet solution in the latter liquid is obtained.

Add some to a dilute solution of potassium permanganate—the latter is decolorized.

Dip a piece of filter paper into a solution of lead acetate, expose it to sulphuretted hydrogen gas until it is converted into black lead sulphide, dry it and then place in a solution of hydrogen peroxide. The sulphide will be oxidized to white lead sulphate.

*3. Estimation of Amount in Solution by titrating against Potassium Permanganate.* Take 10 c.c. of a solution of hydrogen peroxide in a flask, add excess of dilute sulphuric acid, and run in from a glass-stoppered burette a *standard* solution of permanganate, till a pink colour is *just* permanent.

A suitable strength of permanganate is one known as N/10, i.e. a solution containing 3·163 grams per litre.

From an examination of the equation representing the reaction as given on the previous page, it will be seen that 316·3 grams react with 170·1 grams of hydrogen peroxide to form 160 grams of oxygen.

Now N/10 permanganate contains .003163 grams of  $K_2MnO_4$  per c.c., and suppose the volume required =  $v$  c.c.,

Then amount of  $H_2O_2$  in 1 c.c. of original solution =

$$\frac{170\cdot1 \times .003163 \times v}{316\cdot3} = .0001701 \times v \text{ grams.}$$

Perform the experiment three times and find the average. A 10-volume solution should contain about .03 grams per c.c.

*4. Estimation by collecting the Oxygen evolved when mixed with acidified Permanganate.* Use the apparatus shown in Fig. 188. F is a 4 oz. flask into which is put a measured volume of hydrogen peroxide solution, say 10 c.c., and an equal quantity of dilute sulphuric acid.

G is a burette which contains a fairly strong solution of potassium permanganate, and which can be run into F as desired, by means of a glass tap.

E is a tube leading from F to a gas-collecting apparatus B made out of an inverted 100 c.c. burette. D is a swivel made from the neck of a broken Wurtz flask, in which works the gauge tube C.

*To experiment:—*

Put peroxide solution and acid into F, and permanganate into G, taking care that the teat of the burette is filled.

Fill B with water and fit up as shown in diagram. Open tap T to adjust pressure, and read the level of water at A.

Run in permanganate from G until the solution in F is permanently pink. Readjust gauge tube C so that the top is level with the water surface in B.

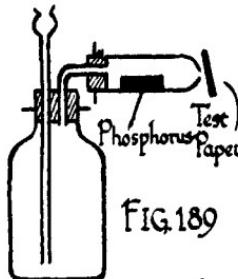
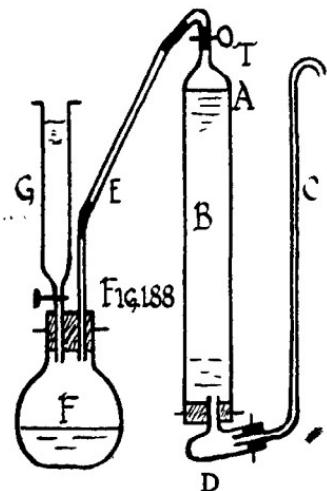
Determine, by reading the new level, the volume of water expelled from B, subtract volume of liquid run in from G.

This gives volume of oxygen obtainable by using 10 c.c. of hydrogen peroxide ; calculate for 1 c.c.

**Ozone.**  $O_3$  or  $O_2O$  or  $O \begin{smallmatrix} O \\ | \\ O \end{smallmatrix}$  is a gas found in small quantities in

the atmosphere in certain districts. Its formation is said to be due to electrical action. Ozone is easily produced from oxygen or air, the process being termed ozonization, e.g. :—

1. An electrical machine working in air yields an amount that is easily recognized by the sense of smell.



2. If air be passed slowly over freshly scraped and moist yellow phosphorus, and the gas tested as it issues, it will be found to be ozonized (Fig. 189).

3 Oxygen is passed through a glass vessel the outside and inside of which are respectively connected to the terminals of an induction coil. Fig. 190 shows Ostwald's form, in which connexion is made to the coil by platinum wires dipping into dilute sulphuric acid. Copper wires will also serve the purpose if cleaned after previous use.

Fig. 191 shows a Siemen's ozone tube, and Fig. 192 a simple modification of the same.

Ozone is also produced :—

1. When dilute sulphuric acid is electrolysed, especially if the current be strong and the electrodes made of thin platinum wire.
2. When a red-hot platinum spiral is suspended in ether vapour.
3. When manganese dioxide, or potassium permanganate, or

barium peroxide is acted upon by sulphuric acid to produce oxygen, ozone is always liberated.

#### PROPERTIES

The chemical properties of ozone are very characteristic, although in some cases they are very analogous to those of hydrogen peroxide.

It has a penetrating and rather unpleasant odour somewhat resembling chlorine ; when heated it is decomposed into oxygen ; it is slightly soluble in water (0·45 per cent. by volume), condenses to a liquid at  $-181^{\circ}\text{C}.$ , and in that condition it is highly explosive. It has powerful oxidizing and bleaching properties due to the ease with which it decomposes to liberate oxygen in an atomic condition.

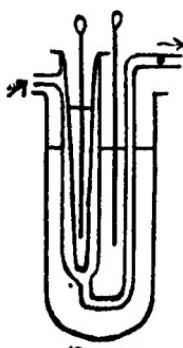


FIG. 190

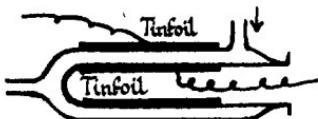


FIG. 191

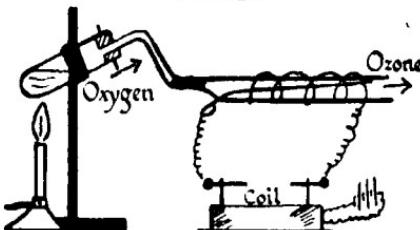
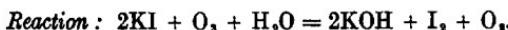


FIG. 192

#### LABORATORY EXERCISES WITH OZONE

1. Investigate its action on a solution of potassium iodide.
2. What happens when "starch iodide" paper is brought into contact with it ?
3. Perform the Houzeau test. Take a piece of litmus paper which has been made *faintly* acid with very dilute nitric acid, and dip it into a solution of potassium iodide. Expose it to ozone. It is *immediately* turned blue.



The caustic potash (KOH) is alkaline, and this turns the red litmus blue.

4. Pass ozonized oxygen through a piece of rubber tubing. The gas which emerges has lost its ozone. Why ?
5. Pass some through a heated glass tube. What happens ?

6. Shake some up with a globule of mercury and note that "tails" are produced. Can you explain this?

*Estimation of the Percentage of Ozone in a Sample of ozonized Oxygen.*

Brodie's method: The apparatus used is a special form of pipette shown in Fig. 193.

Its capacity between  $m$  and  $m'$  is known. It is first filled with strong sulphuric acid by opening tap  $a$ , and closing tap  $b$ , and the pipette put in a vessel containing water at a definite temperature. The end near  $a$  is connected to the supply of ozonized gas and sufficient drawn in to fill the pipette to the mark  $m$ . Tap  $a$  is closed, a vessel containing a solution of potassium iodide is placed under the

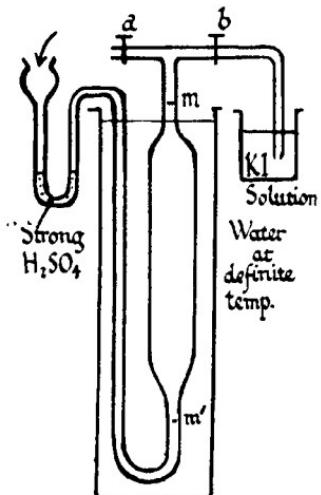


FIG. 193

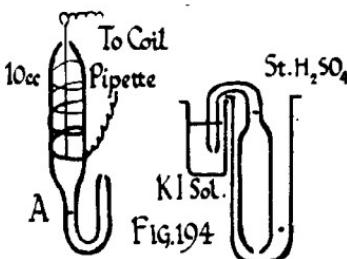


FIG. 194

other end of the pipette, and tap  $b$  opened. The gas from  $m'$  to  $m$  is now forced through the iodide solution, and finally the iodine liberated by this volume estimated.

A simpler and equally efficient apparatus is shown in Fig. 194, which is made by bending the stem of a pipette.

Ozonized oxygen may be made in it (A), or passed into it. To estimate the ozone, it should be inverted in a wide test tube containing strong sulphuric acid, and whilst it is slowly depressed in this liquid, the other end should be immersed in a solution of potassium iodide. When the strong sulphuric acid reaches the graduation mark the volume of gas denoted by the capacity of the pipette has been bubbled through the iodide solution. The free iodine is then estimated by means of a standard solution of sodium thiosulphate.

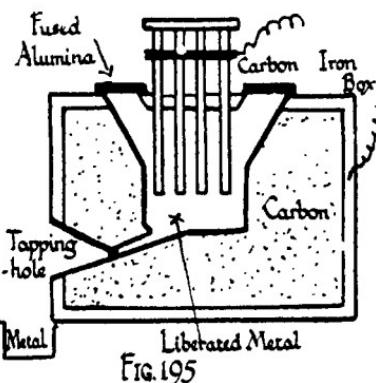


## SECTION XII

### ALUMINIUM

**I**T has been estimated that this element forms one-eighth of the earth's crust, but although so plentiful only one or two of its compounds can, up to the present, be successfully worked for the metal—and these are by no means the most plentiful.

The metal is now prepared entirely by electrolysis. The electrolyte is a fused mass of cryolite, fluorspar, and alumina. Fig. 195 represents in principle the construction of the cell, the temperature of which is nearly  $900^{\circ}\text{C}$ . The oxygen which is liberated combines with the



carbon of the electrodes to form carbon monoxide, the metal sinking to the bottom of the chamber, from which it is periodically tapped.

The alumina,  $\text{Al}_2\text{O}_3$ , is the portion of the electrolytic liquid which is decomposed, the fluorspar and cryolite acting as the flux and solvent.

The annual production of aluminium has increased enormously during the last twenty or twenty-five years, and the price has rapidly fallen. Whereas in 1855 the commercial quality cost £3 10s. per oz. and was a chemical curiosity, it is now (normally) 6d. a lb.

## PROPERTIES

It is a "tin white" metal of great tensile strength, very ductile and malleable, extremely sonorous, and has a low sp. gr. (2·6); its melting-point is 625° C.

It does not tarnish in ordinary air at ordinary temperatures, but burns when strongly heated to form a white oxide called alumina ( $\text{Al}_2\text{O}_3$ ). Nitric acid has not much action on it, but it is dissolved by hydrochloric to form the trichloride.

Aluminium is much more readily attacked by alkalis, particularly caustic soda, potash, and washing soda. A solution of common salt will act upon it, and so will organic acids in the presence of this compound—consequently alkaline liquids must not be boiled in aluminium vessels.

It is a powerful reducing agent, and is used in the "Faermit" process in the powdered condition for reducing oxides of iron, manganese, chromium, etc., to the metallic condition in small welding operations.

Several important alloys are made from it, of which the best known are aluminium bronzo (90 per cent. copper, 10 per cent. aluminium) and magnalium (90 per cent. aluminium, 10 per cent. magnesium, etc.).

*Uses.* For metallic parts of airships, aeroplanes, balances, cooking utensils, surgical instruments, paint, reducing agent, ornamental and decorative purposes.

In some ways its use is restricted by the difficulty experienced in soldering it—no really satisfactory method of doing this has been invented yet.

## Chief Compounds of Aluminium.

1. The Alums. These are double sulphates; the potassium salt was one of the earliest compounds of the metal prepared.  $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ .

2. Clay. Kaolin, or China clay, contains a large percentage of the metal. Its composition is  $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 24\text{H}_2\text{O}$ . Clay of all kinds consists essentially of silica and alumina in varying proportions, associated with smaller quantities of lime, magnesia, oxides of iron, and alkali metals.

3. Cryolite.  $3\text{NaF} \cdot \text{AlF}_3$ . Largely used as a flux in certain metallurgical operations.

4. Aluminium oxide,  $\text{Al}_2\text{O}_3$ —alumina. In a natural condition it occurs associated with small quantities of other metallic oxides as bauxite, corundum, emery, ruby, amethyst, sapphire, topaz, turquoise.

5. Aluminium trichloride,  $\text{Al}_2\text{Cl}_6$ , used for carbonizing cotton in mixtures of cotton and wool, is prepared in the anhydrous condition

by passing chlorine gas over heated aluminium foil, and for ordinary purposes by the action of strong hydrochloric acid on the metal and concentrating the solution.

6. Other salts used for textile purposes are the acetate, made by dissolving the hydroxide in acetic acid, or by the addition of lead' or calcium acetate solution to a solution of aluminium sulphate. The impure commercial acetates of aluminium are used by dyers and calico-printers as mordants for alizarine reds, and on that account are known in trade as "red liquor."

Aluminium acetate is a very efficient "shower-proofing" chemical for cotton or wool cloth. The material to be treated is put for some hours in a warm solution of the salt (strength 8°-10° Twaddell), then passed through a soap solution containing 50-75 grams of soap per litre at a temperature of about 45° C., dried in a hot chamber and calendered. Japan wax, gums, oils, paraffin, wax, etc., are sometimes added to the soap bath.

*Preparation of Alum.*

- 1. In the laboratory, by adding aluminium sulphate to potassium sulphate in proper proportions and crystallizing from the hot solution.
- 2. From alum stone— $\text{Al}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 2\text{Al}_2\text{O}_3 \cdot 8\text{H}_2\text{O}$ . The material is calcined and then the liquid lixiviated with hot water when the  $\text{Al}_2\text{O}_3$  remains undissolved. The alum may be crystallized out after sedimentation. Or, the calcined mass is treated with sulphuric acid to dissolve the oxide and then before crystallization the requisite amount of potassium sulphate is added.

The former method produces what is called Roman alum. The iron present as an impurity may be separated by filtration and recrystallization.

3. From alum shale, which is a rocky mass consisting of aluminium silicate and iron pyrites. It is first roasted and exposed to air and moisture. The pyrites is oxidized and sulphuric acid is formed, which acts upon the shale, making aluminium sulphate. The mass is lixiviated, concentrated, and potassium chloride added. The iron sulphate which has also been formed is decomposed to chloride, and potassium sulphate produced. When concentrated it is well stirred to ensure the precipitation of the alum in small crystals—the product being known as "meal."

4. From bauxite ( $\text{Al}_2\text{O}_3$ ). This mineral is roasted, treated with sulphuric acid, and lixiviated with water. The solution is concentrated, potassium chloride added and then crystallized.

5. From clay. Purified and calcined China clay is boiled with oil of vitriol for several hours, then with several times its weight of water till it makes a syrup. It is filtered and cooled, the correct amount of potassium sulphate added and then crystallized.

**Alum** is very soluble in hot water, but only slightly in cold :—

100 grams of water at 0° C. dissolve 3·9 of alum.

" " " 50° C. " 44·1 "

" " " 100° C. " 357·5 "

It is insoluble in alcohol; when heated it melts and dissolves in its own water of crystallization, which is gradually expelled, until at a dull red heat a non-crystalline and anhydrous substance is obtained called *burnt alum*, which is much less soluble in water than the crystalline form.

The chief use for alum is as a *mordant* in dyeing. When sodium carbonate or sodium hydrate is added to alum solution, till the precipitate first formed is redissolved, a *basic alum* (called neutral alum) is formed. This compound very readily gives up alumina to fibres impregnated with it. If the fibres coated with this mordant be now passed through solutions of certain colouring matters, the two unite to form a coloured "lake" which is not removed by boiling water.

**Aluminium sulphate**, also known as cake alum, patent alum, concentrated alum, and in the impure condition containing considerable quantities of iron, as alumino-ferric, is prepared by dissolving aluminium hydroxide, bauxite, or clay in sulphuric acid. It has an acid reaction and is used instead of alum for many purposes.

**Aluminium hydroxide** is formed as a white gelatinous precipitate when caustic alkalis are added to solutions of aluminium salts. It has considerable application as a precipitating and clarifying agent.

### ZINC

Zinc, its alloys and compounds, are of great practical importance. The metal itself is used in large quantities for coating sheet iron (so-called galvanized iron), and in the powdered condition as a reducing agent for indigo.

Brass, bronze, and German silver—in all of which zinc is present—have a very wide application. Zinc oxide is used as a pigment, and zinc chloride is the most generally used antiseptic in the cotton industry. Other important compounds are white vitriol, or zinc sulphate, zinc carbonate, and zinc sulphide.

The ores used for the extraction of the metal are :—

1. Calamine or zinc carbonate.
2. Blende, black jack or zinc sulphide. (The colour of this ore is due to the presence of sulphide of iron as an impurity.)

3. Red zinc ore—an impure oxide.

**Extraction.** The ore is first calcined to convert it into the oxide, and then it is mixed with carbon and strongly heated in retorts, when the oxide is reduced first to the metallic condition and then vaporized. The gaseous metal is condensed in receivers.

In the Belgian process the retorts are small, being about 3-4 ft. long and 9 in. in diameter, as many as 80 being put in one furnace, each holding a charge of about 40 lb.

The Silesian furnace contains about 30 retorts,  $\square$  shaped in section, and are much larger than those used in Belgium, each holding a charge of 5 cwt.

The condensing-chambers are attached to the mouths of the retorts as shown in Fig. 196.

The metal so obtained is called "spelter" and is very impure—lead, iron, tin, antimony, arsenic, copper, cadmium, magnesium, and aluminium may all be present.

It may be purified by redistillation, but a better product is obtained if it is dissolved in acid, the carbonate produced by precipitation, and this compound reduced with charcoal made from sugar.

Zinc is very malleable at a temperature of  $121^{\circ}$  C., but when heated to  $204^{\circ}$  C. it again becomes brittle and can be powdered up in a mortar.

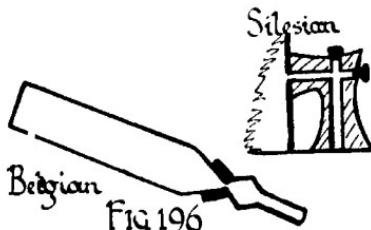
When exposed to air it is slowly attacked to form zinc oxide, which gradually changes to the carbonate; and this layer when completely formed over the surface protects it from further oxidation.

The metal is very soluble in dilute acids, alkalis, and slightly in boiling water or steam. The salts of zinc so formed are very poisonous, and therefore zinc vessels are not suitable for cooking utensils. Galvanized cisterns for storing water are on that account objectionable unless they be coated finally with a layer of tin.

Zinc chloride is a very important textile chemical, and is made by dissolving all sorts of waste zinc, ashes and skimmings, in hydrochloric acid. It can also be made by dissolving calamine in the same acid.

Commercial zinc chloride is liable to contain several impurities, the methods for detecting which are given in Section XV, pages 188-189. The manufacturer endeavours to produce a product free from acid and iron. For use in this country it is usually sold as a strong solution in water—about  $100^{\circ}$ - $104^{\circ}$  Twaddell, but for export purposes it is further concentrated till it sets as a solid on cooling and contains up to 95 per cent. of anhydrous zinc chloride.

The substance known in the cotton trade as "zinc," or "antiseptic" is zinc chloride. Occasionally the term "anti" is used to denote magnesium chloride.



By boiling a solution of zinc chloride of sp. gr. 1.7 with excess of zinc oxide, a basic or oxychloride is obtained which dissolves silk and is used to estimate that material when mixed with wool and vegetable fibres.

Zinc chloride is very hygroscopic, and a very efficient "fungicide" for cotton. It is very soluble in water, slightly in alcohol; has a melting-point of 250° C., and a boiling-point of 730° C., but while being heated to that temperature, particularly if water be present, considerable decomposition results, hydrochloric acid being evolved with reduction to oxychloride.

Zinc sulphate, known in the crystalline condition as "white vitriol,"  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ , can be made by dissolving the metal in dilute sulphuric acid, or roasting the ores and then treating them with the acid and recrystallizing.

It has some application in dyeing and calico-printing; it is used in the tanning industry as a preserving and clarifying agent, as an astringent in eye "lotions," and as a dryer for boiled oil when used in paint.

Zinc oxide and zinc sulphide are both used as pigments. The former can be prepared by burning the metal in air, and the latter by precipitating from solutions of the chloride or sulphate with sulphuretted hydrogen in alkaline solution, or heating an intimate mixture of zinc dust with half its weight of powdered sulphur.

Compounds of zinc heated on charcoal in an oxidizing flame give a white infusible residue. If this be allowed to cool and a few drops of cobalt nitrate solution dropped on it and the mass again heated, a very distinctive green residue is formed.

#### MAGNESIUM AND ITS CHIEF COMPOUNDS

The element itself does not occur free in nature, but it is present in several minerals, the most important being:—

1. Magnesite or magnesium carbonate,  $\text{MgCO}_3$ .
2. Dolomite or magnesium limestone—mixed carbonates of magnesium and calcium.
3. Kieserite—magnesium sulphate,  $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ .
4. Carnallite—magnesium and potassium chlorides:—  
 $\text{MgCl}_2 \cdot \text{KCl} \cdot 6\text{H}_2\text{O}$ .
5. Epsom salts—magnesium sulphate,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ .
6. Various silicates, e.g., talc, hornblende, asbestos, olivine, meerschaum, serpentine.

The metal is prepared by the electrolysis of the fused chloride. A temperature of 700° C. is obtained by surrounding an iron crucible with burning "gaseous fuel." The crucible is the cathode. The

anode is a stout carbon rod, surrounding which is a porous cylinder to convey away the liberated chlorine.

#### PROPERTIES OF THE METAL

Sp. gr. 1.75; melting-point, 632° C.; boiling-point, 1,100° C. It is a silver-white metal, ductile at high temperatures and fairly malleable. It oxidizes slowly in *moist* air, but not in dry air or oxygen. Heated in air, it burns, giving a dazzling white flame rich in chemical rays. On this account it is used as an artificial illuminant in photography, but, owing to the production of a white smoke of magnesium oxide, it cannot be used long at a time. Burning magnesium is often employed to examine and compare dyed fabrics for shade.

Heated in steam, it decomposes it, forming the oxide and liberating hydrogen. Lighted, and plunged into carbon dioxide, it continues to burn, decomposing the gas with liberation of carbon and formation of magnesium oxide. Heated in nitrogen, it combines with it to form a nitride,  $Mg_3N_2$ . This was one of the earlier methods adopted for the isolation of argon from the atmosphere.

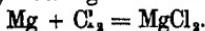
It is very soluble in dilute acids and solutions of ammonium salts, with liberation of hydrogen, this occurring even with nitric acid if it be sufficiently dilute and a few inches of magnesium ribbon used. It is insoluble in caustic potash or soda. It is a powerful reducing agent at high temperatures.

The chief compounds of magnesium are the chloride, oxide, sulphate, and carbonates.

*Magnesium chloride* ( $MgCl_2$ ) can be prepared :—

1. From the natural carnallite by fractional crystallization. Magnesium chloride is much more soluble than potassium chloride, and thus remains in the mother liquor after most of the latter has been deposited. The crystals when formed by further concentration have the composition  $MgCl_2 \cdot 6H_2O$ , and are very deliquescent.

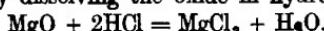
2. By burning the metal in chlorine :—



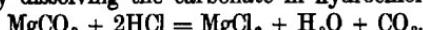
3. By dissolving the metal in hydrochloric acid :—



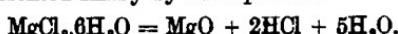
4. By dissolving the oxide in hydrochloric acid :—



5. By dissolving the carbonate in hydrochloric acid :—



To obtain the anhydrous salt, concentration of the solution to dryness will not suffice, as it undergoes a series of chemical changes which are represented finally by the equation :—



Although the anhydrous chloride is never required for textile purposes, this reaction is very important because it explains what happens in "singeing" when a cloth contains magnesium chloride. In this case it is the liberated hydrochloric acid which, acting on the cotton, converts it into hydrocellulose and so produces tendering of the fabric.

If ammonium chloride be added to a solution of magnesium chloride, a double salt is formed,  $MgCl_2 \cdot NH_4Cl \cdot 6H_2O$ . When this is heated it is first dehydrated, and then the ammonium chloride volatilizes, leaving the anhydrous magnesium chloride as a fused mass, which congeals to a white crystalline solid.

Magnesium chloride is a very deliquescent substance, and on this account it is largely used as a sizing ingredient, particularly in the "heavy trade." Its use is attended with considerable danger unless zinc chloride or some equally efficient antiseptic be used with it, as it has no antiseptic properties whatsoever. The substance is often known under the name of "anti"—a most unfortunate term, as it tends to convey the impression that it has preservative properties.

*Magnesium oxide or magnesia* ( $MgO$ ) is obtained by :—

1. Burning magnesium in air or oxygen.
2. Calcining magnesium nitrate ( $MgNO_3$ )<sub>2</sub> =  $MgO + 2NO_2 + O$ .
3. Calcining the carbonate or basic carbonate :—



4. Converting the chloride into carbonate, and then gently igniting the dried powder. If excess of sodium carbonate is added and the mixture well boiled, the composition of the carbonate produced is  $2MgCO_3 \cdot Mg(OH)_2 \cdot 2H_2O$ . On ignition this becomes



The hydroxide is obtained by dissolving the oxide in water. Solubility of the oxide is about 1 in 55,000 of cold water, and less in hot.

*Magnesium sulphate* ( $MgSO_4$ ) occurs naturally in the Stassfurt deposits as kieserite,  $MgSO_4 \cdot H_2O$ . Upon treating this with water and recrystallizing, the pure salt is obtained as  $MgSO_4 \cdot 7H_2O$ . It is present in many mineral springs, and from its occurrence in one of them has been named Epsom salts.

When the carbonate is treated with dilute sulphuric acid, the following reaction occurs :—



If magnesium limestone is used, sulphates of lime and magnesia are both formed. The former, being insoluble, may be removed by sedimentation, but obtained in this way the magnesium sulphate is not so pure as that made from kieserite.

The salt has a bitter taste ; it is completely dehydrated at 200° C. ; its solubility at ordinary temperature is 126 in 100 of water. It has a considerable application in medicinal saline mixtures, and in textiles it is used as a finishing material in certain kinds of finishes for cotton cloth. For this purpose it is advisable that it should be *free from traces of magnesium chloride*, as the presence of the latter may lead to partial solution of the sulphate when the cloth is in a humid atmosphere. When the cloth becomes drier, the sulphate recrystallizes in the fibre, which results in tendering of the fabric, particularly if it be repeated once or twice.

*Magnesium carbonate*,  $MgCO_3$ , occurs naturally as magnesite, and as magnesium limestone mixed with calcium carbonate.

It is decomposed by heat much more easily than calcium carbonate to form the oxide with liberation of carbon dioxide.

## SULPHUR

This is an element which occurs in large quantities in nature, both in the free state and in combination.

1. As native sulphur (i.e. sulphur uncombined, but mixed with earthy matter) in all volcanic districts, e.g. Italy, Sicily, Iceland, United States.

2. Forming sulphides with certain metals, it occurs in most ores, e.g. pyrites or iron sulphide, copper pyrites, galena or lead sulphide, zinc blonde, cinnabar or mercury sulphide, etc.

3. Sulphates which contain sulphur combined with a metal and oxygen, e.g. gypsum, alabaster or calcium sulphate, heavy spar or barium sulphate, kieserite or magnesium sulphate, etc.

Sulphur is prepared chiefly from native sulphur, but considerable quantities are also obtained from alkali waste and coal-gas waste.

1. *From native sulphur*. It is liquated where found, i.e. it is stacked on the side of a slope, covered with a turf roof and fired, the entrance of air being reduced to a minimum. Some is burnt, which supplies the heat to melt the rest, which then flows along the sloping floor until it gets outside the stack (Fig. 197). This simple process separates it from a considerable quantity of the earthy matter with which it was mixed. In this condition it is generally shipped. It is afterwards purified by redistillation (Fig. 198).

2. *From alkali waste* (Mond's process). Alkali waste is a mixture of calcium sulphide and calcium oxide. It is suspended in water and oxidized by blowing a current of air through it. This produces such compounds as calcium thiosulphate, calcium polysulphides, calcium hyposulphide, etc., and liberates a large quantity of sulphur. It is alternately oxidized and lixiviated, and finally hydrochloric acid is

added to precipitate the remainder of the sulphur, which is purified as before by distillation.

3. From coal-gas waste, which is hydrated ferric oxide which has absorbed the sulphuretted hydrogen from coal gas. It is exposed to air and moisture, by which means sulphur is liberated. The mass is afterwards distilled.

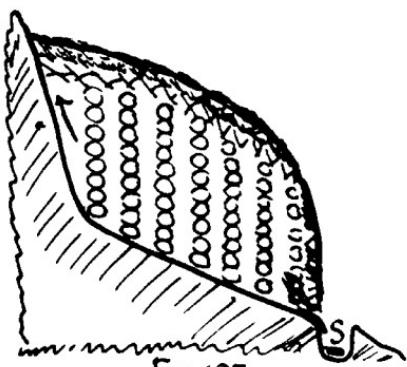


FIG. 197

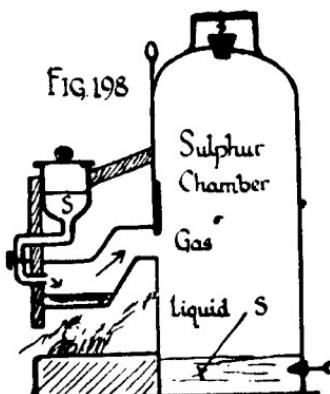


FIG. 198



FIG. 199

4. By mixing sulphur dioxide and sulphuretted hydrogen gases in the presence of water vapour sulphur is precipitated.  
Sulphur can be prepared in at least *four* distinct varieties or *allotropic modifications*.

1. *Rhombic* sulphur, made by dissolving sulphur in carbon disulphide, filtering and allowing the clear liquid to evaporate slowly at ordinary temperature.

2. *Prismatic* sulphur, prepared by carefully melting sulphur in a beaker, allowing it to stand until it has *partly* solidified, and then pouring away the still liquid portion. Prismatic crystals line the sides and bottom of the beaker.

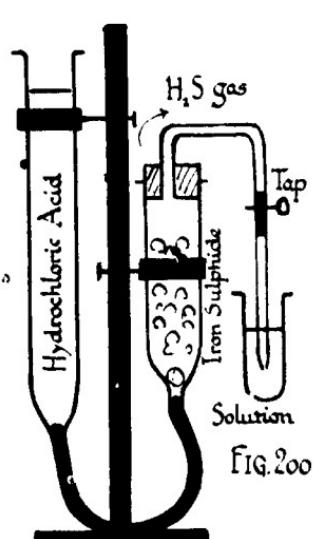
3. *Plastic* sulphur is formed when melted sulphur is poured in a thin stream into cold water (Fig. 199).

4. *White amorphous* sulphur is produced when carbon disulphide is exposed to sunlight, or when hydrochloric acid is added to ammonium sulphide. It is insoluble in carbon disulphide.

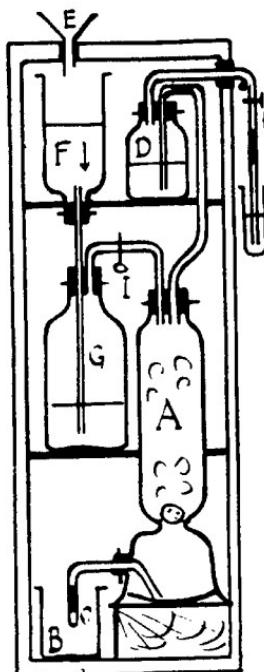
#### PROPERTIES OF THE RHOMBIC AND STABLE VARIETY

Insoluble in water; soluble in carbon disulphide; burns with pale blue flame to form sulphur dioxide; non-conductor of

electricity; bad conductor of heat; yellow in colour; melts at  $114^{\circ}\text{C}$ . to a pale yellow liquid, which is very mobile. Heated still further, the liquid gradually darkens in colour and becomes more and more viscous, until at  $230^{\circ}\text{C}$ . it is almost black and can scarcely be poured from the vessel; heated to a higher temperature, it becomes somewhat less viscous but still remains black, and ultimately



*To Use.*—Put in the acid and iron sulphide. Open the tap—the acid acts on the sulphide and liberates the gas, which passes through the solution. When enough has passed, close the tap; the gas then collects and forces the acid back into its own tube, thus stopping the action. The acid and sulphide tubes are connected by india-rubber tubing.



boils at  $448^{\circ}\text{C}$ . In cooling it goes through similar changes in the reverse order.

Sulphur is used largely in the manufacture of sulphuric acid, ebonite, vulcanite, matches, gunpowder, dye-stuffs, sulphides, etc., and also as a fungicide and insecticide.

Hydrogen sulphide, or, as it is commonly termed, sulphuretted hydrogen, is by far the more important of the two compounds which sulphur forms with hydrogen. It is found dissolved in certain natural mineral waters, e.g. at Harrogate, and it is evolved from active volcanoes.

*Preparation :—*

1. If the *pure* gas be required, antimony trisulphide is treated with strong hydrochloric acid.
2. For ordinary purposes ferrous sulphide (made by melting powdered sulphur with iron filings until combination results) is acted upon with dilute sulphuric acid or moderately strong hydrochloric acid in one or other of the forms of apparatus shown in Figs. 200–203.

The correct method of using each form is given under its own illustration.

In Fig. 201 the iron sulphide is placed in the vessel A, and water in the wash bottle D. Hydrochloric Acid (1 : 1) is poured through the

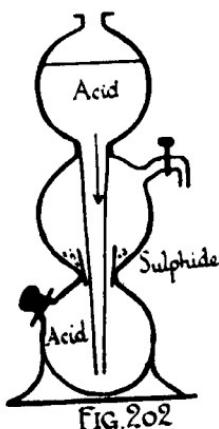


FIG. 202

— Acid is put in the top bulb, from which it passes to the bottom and then to the centre. As soon as it reaches the sulphide the tap should be closed. As the gas accumulates the acid is forced back.

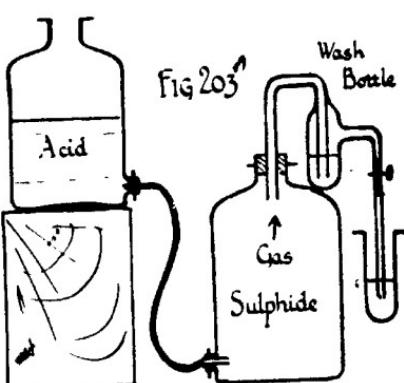


FIG. 203

When in use the aspirator containing the acid should be placed on a block of wood.

After using the gas the tap should be closed, then the aspirator containing the acid put on the bench and the one containing the sulphide put in its place.

funnel E and into the reservoir F until the bottle G is filled, and the acid begins to fall on the sulphide in A. The evolved gas passes up through the water in D, and is drawn off at the tap H, which is placed outside the cabinet, away from the working parts of the apparatus. When the tap is closed the acid is driven into the bottle G, and thence to the reservoir F. Communication between G and A can be stopped by the clip I.

Used acid is withdrawn from A into the basin B by removing the glass rod C.

3. It may also be prepared from its elements by direct union, by passing a stream of hydrogen and sulphur vapour through a strongly heated tube.

4. The gas is also produced when organic matter containing sulphur decays, e.g. eggs. When coal is distilled, sulphuretted hydrogen is evolved, and on that account the coal gas is "scrubbed" before it reaches the gasometer.

### PROPERTIES

Colourless gas; extremely foetid smell and very poisonous if breathed into the lungs. It is on this account that the gas is generated in special forms of apparatus, which should be kept (and used) in a fume chamber if possible.

It is soluble in water to the extent of about 3 in 1 at ordinary temperatures. Its solution is acid to litmus, and decomposes after a time on exposure to air—sulphur being precipitated.

The gas burns with a bright blue flame, producing sulphur dioxide and water. It forms an explosive mixture with oxygen when mixed in the proportion of 2 to 3.

When passed into strong sulphuric acid it is decomposed—sulphur dioxide, water, and sulphur being formed. It is absorbed by lime, but calcium chloride has no action on it.

In contact with metals, or when passed into metallic solutions, it produces *sulphides*, e.g. tin, lead, silver, etc. The "lead reaction" is used as a test for the gas.

The chief use for hydrogen sulphide is as a laboratory reagent. Its reactions in this respect form the basis of the ordinary methods of analytical chemistry.

It is also used—generally in the form of ammonium sulphide—for "oxidizing" (sic) copper and silver in art metal work.

*Preparation of Hydrogen Sulphide—experiments illustrating its Properties. Its action on metallic Solutions in the Formation of Sulphides.*

Arrange the apparatus shown in Fig. 200.

Place ferrous sulphide in left-hand vessel and hydrochloric acid—half strong, half water—in the other.

Open the clip; the acid flows amongst the iron sulphide and evolves sulphuretted hydrogen.

*Tests.* 1. Is it soluble in water?

2. Describe its smell.

3. Does it burn? Does it support combustion?

4. Action on lead acetate paper.

Note its action on "metallic solutions."

Pass a stream of sulphuretted hydrogen through each in turn.

*Note.* 1. Whether a precipitate of a sulphide is produced, i.e.

Is it soluble or insoluble in water?

2. The colour of the precipitate (if there be one).

*Test :—*

- Filter off,  
wash, and divide  
the precipitate } into five parts in  
separate T.T.'s.
3. Its solubility in hydrochloric acid.  
4. „ „ ammonia.  
5. „ „ nitric acid.  
6. „ „ ammonia disulphide.  
7. „ „ ammonium chloride.

Carefully record *all* your results.

Note what a large number of metallic solutions yield sulphides when treated with hydrogen sulphide, and the great similarities when treated with certain reagents. There are also many points of difference when individually considered, especially in regard to colour.

It is on this account that the reactions of the sulphides of the metals are most often used as the basis of analytical chemistry.

#### SOME OF THE PRINCIPLES OF ANALYSIS

I. Sulphides which are *insoluble in hydrochloric acid*—that is, if a solution of any of these metals be first acidulated with hydrochloric acid and then the gas passed through: these sulphides will be precipitated :—

Mercury (ous, ic) silver, lead (partly), copper, cadmium, bismuth, tin (ous and ic), antimony, and arsenic (often called the copper group). *All the rest of the sulphides are soluble either in hydrochloric acid or water.*

II. Sulphides which are *soluble in hydrochloric but insoluble in ammonium hydrate* :—

Iron (ous and ic), chromium, aluminium, nickel, cobalt, zinc, manganese, magnesium. (Iron and zinc group.)

III. Sulphides which are *soluble in acid, alkali, and water* :—

Barium, strontium, calcium, potassium, sodium, ammonium.

IV. Silver, mercury (ous), and lead (partly) are precipitated as chlorides when the hydrochloric acid is added to the solution. (Silver group.)

It is also possible to subdivide the groups. Thus—

*In the copper group* : arsenic, antimony, and tin sulphides are all soluble in ammonium disulphide. (Arsenic sub-group.)

*In the iron and zinc group* : The hydrates of iron, chromium, and aluminium are insoluble in water. Therefore when ammonium hydrate is added to solutions of salts of these metals, a precipitate is produced before sulphuretted hydrogen is passed.

Again, magnesium sulphide is not precipitated in the presence of ammonium chloride. Its phosphate is insoluble in water.

*In Group III* a division may be made by precipitating barium, strontium, and calcium as carbonates—which are all insoluble in water. (Barium group.)

These operations are generally referred to as *Grouping*.

*The order of working* will therefore be as follows :—

1. Prepare a solution of the salt in water.
2. Add enough hydrochloric acid to make the solution acid to litmus paper.

*A precipitation indicates presence of silver group.* (Silver, mercury (ous), lead.)

*No precipitation indicates the absence of silver group.*

3. Filter off the precipitate (if there be one).
4. Pass through the solution (or filtrate) sulphuretted hydrogen gas.

*A precipitation indicates presence of copper group.* Filter off and test its solubility in ammonium disulphide.

If insoluble—absence of arsenic sub-group.

If soluble—it is arsenic, tin, or antimony.

Insoluble in ammonium disulphide, and is :—

(a) Black—mercury (ic), lead, or copper.

(b) Brown—bismuth.

(c) Yellow—cadmium.

*No precipitation with sulphuretted hydrogen—shows absence of copper group.*

5. Boil the solution (or filtrate) for a few minutes, add a few drops of nitric acid, and boil again. (This is to get rid of the hydrogen sulphide and oxidize the iron.).

6. Add ammonium chloride and then ammonium hydrate.

*A precipitation—presence of iron sub-group* (iron, chromium, aluminiunm).

*No precipitation—absence of iron sub-group.*

7. Filter off the precipitate (if there be one).

8. Pass through the solution (or filtrate) sulphuretted hydrogen.

*A precipitation indicates presence of zinc sub-group.*

(a) Zinc—white precipitate.

(b) Manganese—flesh-coloured precipitate.

(c) Nickel and cobalt—black precipitate.

*No precipitation—absence of zinc sub-group.*

9. Filter off precipitate (if there be one).

10. Add to solution (or filtrate) ammonium carbonate.

*A precipitation—barium, strontium, or calcium carbonates.*

*No precipitation—absence of barium group.*

11. To filtrate or solution add sodium phosphate, when magnesium phosphate will be precipitated if a magnesium salt were present.

12. Potassium, sodium, and ammonium are identified separately and by other means.

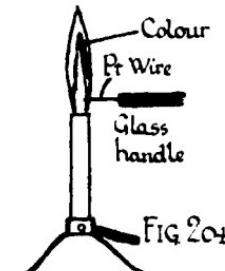
In order that the student shall be able to apply simple tests to identify textile chemicals and mill stores it is very advisable that he

should have some knowledge of analytical processes, and a suitable exercise at this stage is the analysis of a simple salt.

#### SCHEME FOR THE ANALYSIS OF A SIMPLE SALT

##### Preliminary Tests.

1. Heat the substance alone in a *small* dry test tube.  
Odour of sulphur dioxide—hydrosulphuric or sulphurous acid.  
Evolution of carbon dioxide—carbonic acid.  
Metallic sublimation—mercury.  
Yellow hot, white cold—zinc.
2. Treat with dilute hydrochloric acid, and determine the nature of the gas or vapour evolved (see page 52).

3. Treat with strong sulphuric acid.
  4. Treat with strong sulphuric acid and lead peroxide.
  5. Put a grain or two in a watch-glass, add strong hydrochloric acid; dip into it a platinum wire, and find the "flame reaction." (For method of holding the wire in the colourless bunsen flame, see Fig. 204.)
- |   |  |
|---|--|
|  | Green—barium, copper, boric acid.<br>Yellow—sodium.<br>Violet—potassium (red through indigo prism or cobalt glass).<br>Red—calcium (lime).<br>Crimson—strontium. |
|---|--|

6. Make a borax bead by fusing some borax on the end of a platinum wire. Fuse in a *minute* portion of the substance and note the colour of the bead.

Blue—cobalt, copper.

Violet—manganese.

7. Fuse a little on charcoal in the reducing flame of a blowpipe. Beads or scales of metal—silver, copper, iron, cobalt, nickel, tin, lead, bismuth, and antimony.

8. Fuse some more on charcoal in oxidizing flame. Cool, and then moisten the residue with a solution of cobalt nitrate. Reheat in the same flame—

Blue residue—aluminium.

Green residue—zinc.

Pink residue—magnesium.

9. Prepare a solution (a) in water, or if insoluble (b) nitric acid or (c) hydrochloric acid. (d) If not soluble in water or dilute acid, fuse with fusion mixture, and then boil the "melt" with water. Filter. In the filtrate examine for acids, and test the residue, after dissolving in hydrochloric acid, for bases.

*To detect the Basic Radicle :—*

1. To one portion of the solution add a solution of caustic soda.
  - (a) White precipitate *soluble* in excess—lead, zinc, antimony, aluminium, tin.
  - (b) White precipitate *insoluble* in excess—bismuth, cadmium, magnesium, calcium, barium, strontium, manganese (darkens).
  - (c) Yellow precipitate—mercury (ic).
  - (d) Black precipitate—mercury (ous).
  - (e) Blue precipitate—copper, cobalt.
  - (f) Dark brown precipitate—silver.
  - (g) Dirty green precipitate—iron (ous).
  - (h) Reddish brown precipitate—iron (ic).
  - (i) Green soluble in excess—chromium.
  - (j) Green insoluble in excess—nickel.
  - (k) Evolution of ammonia gas—ammonium.
  - (l) No precipitate—arsenic, potassium, sodium, ammonium.
2. To another portion add hydrochloric acid.  
 White precipitate—lead, mercury (ous), silver. Wash this precipitate with warm ammonia.  
 No change—lead.  
 Dissolves—silver.  
 Blackens—mercury.
3. If no precipitate has been produced with the acid, to the *same* solution add sulphuretted hydrogen gas.  
 Black precipitate—mercury (ic), lead, copper.  
 Dark brown precipitate—bismuth, tin (ous).  
 Yellow precipitate—cadmium, arsenic, tin (ic).  
 Brick red precipitate—antimony.
4. If no precipitate in Nos. 1 and 2, to a *fresh* portion of solution add ammonium chloride and ammonia.  
 Dirty green precipitate—iron (ous).  
 Reddish brown precipitate—iron (ic).  
 Green precipitate—chromium.  
 White precipitate—aluminium.
5. If no precipitate in No. 4, to the same solution add ammonium sulphide.  
 Black precipitate—nickel, cobalt.  
 White precipitate—zinc.  
 Buff precipitate—manganese.
6. If no precipitate in No. 5, to the same solution add ammonium carbonate solution.  
 White precipitate—barium, strontium, calcium (distinguish by flame reactions).

7. If no precipitate in No. 6, boil down the same solution, add more ammonia and then sodium phosphate.

White crystalline precipitate—magnesium.

To detect the Acidic Radicle:—

Test solution with litmus. If acid, neutralize with ammonia (any precipitate may be filtered off and neglected); if alkaline, neutralize with nitric acid.

1. To some of the *neutral* solution add silver nitrate. If a precipitate is produced, divide it into two parts.

(a) Try the effect of heat on one part.

(b) Determine if soluble or insoluble in nitric acid with the other part.

If the precipitate be *soluble* in nitric acid and is :—

(a) White, rapidly darkening—it may be thiosulphuric.

(b) White, darkened by heat—it may be sulphurous, boric, carbonic.

(c) White, dissolved on heating—it may be acetic.

(d) White, unaltered by heat—it may be oxalic, tartaric, citric.

(e) Yellow—it may be phosphoric, arsenious.

(f) Brown—it may be arsenic.

(g) Red—it may be chromic.

If the precipitate be *insoluble* in nitric acid and is :—

(a) White, turns purple—it may be hydrochloric.

(b) White—it may be hydrocyanic.

(c) Yellowish white—it may be hydrobromic.

(d) Yellow—it may be hydriodic.

(e) Black—it may be hydrosulphuric.

Confirmatory tests with the *original* solution should be applied to distinguish :—

*Thiosulphuric*—with hydrochloric acid gives a yellow precipitate and evolves sulphur dioxide.

*Sulphurous*—with hydrochloric acid gives off sulphur dioxide.

*Carbonic*—with hydrochloric acid evolves carbon dioxide.

*Boric*—acidify with hydrochloric, dip in turmeric paper, dry it, the paper turns green.

*Acetic*—warmed with *strong* sulphuric acid—odour of vinegar.

(a) Add calcium chloride solution.

(1) White precipitate in the cold—oxalic, tartaric.

(2) White precipitate on boiling—*citric*.

(b) Heat another portion with strong sulphuric acid.

(1) Blackens—*tartaric*.

(2) No blackening—*oxalic*.

*Phosphoric*—add a little of the original solution to ammonium molybdate solution warm—yellow precipitate.

*Arsenic* or *Arsenious*—put some of the *original substance* in an ignition tube with a small piece of charcoal and heat in bunsen flame—mirror of metallic arsenic sublimes.

*Chromic*—Lead acetate gives a bright yellow precipitate.

<i>Hydrochloric</i>	{ Heated with a peroxide	{ chlorine evolved
<i>Hydrobromic</i>	{ and strong sulphuric	{ bromine evolved
<i>Hydriodic</i>	{ acid	{ iodine evolved

*Hydrocyanic* (Prussian blue test)—add solutions of ferrous sulphate and ferric chloride, then excess of caustic soda. Boil, cool, and acidify with hydrochloric acid.

*Hydrogulphuric*—add hydrochloric acid—sulphuretted hydrogen evolved.

2. If silver nitrate does *not* give a precipitate in the neutral solution all the above-named acids are absent.

• Acidify a fresh portion of the solution with nitric acid, and add a solution of barium nitrate.

White precipitate = *sulphuric acid*.

3. If still no precipitate, test the *original substance* as follows:—

Dissolve in water (all nitrates are soluble in water). To this solution add a few c.c. of strong sulphuric acid, carefully cool the mixture, and when cold, pour on the top of it a strong cold solution of ferrous sulphate. A “brown ring” at the junction indicates *nitric acid*.

## APPLICATION OF CHEMISTRY TO TEXTILES

ALTHOUGH in its narrowest sense the term "textile" refers to the process of weaving only, by convention it has now a much wider significance, and is taken to include other branches of the manufacture of cotton, wool, and silk.

In a similar sense we desire to use the term "textile chemistry," applying it with reference to instruction in the principles of all branches of the industry and particularly to the properties of the materials that are necessary to produce finished cloth from raw fibre.

Textile chemistry in its more advanced form consists of a specialized study of each of the separate processes, and therefore the subject should be continued under the various branches, such as dyeing, sizing, bleaching, etc.

### SECTION XIII

#### THE NATURAL FIBRES

THE chief natural fibres in use in this country for textile purposes are cotton, wool, linen, and silk, of which the first two are by far the more important.

Besides these a considerable amount of artificial silk is used.

The characteristic appearance of fibres can be seen best under the microscope. The principle of the construction of this instrument is illustrated in Fig. 205.

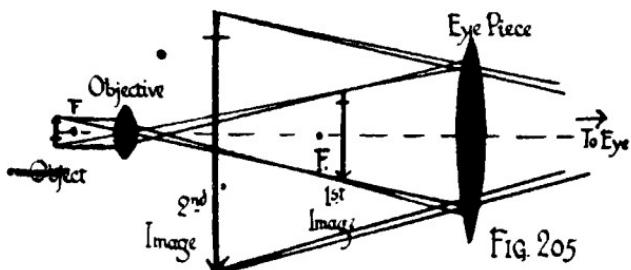
The object under examination is placed just beyond the focus (F) of a lens (called the objective) of short focal length. Rays of light passing through this lens produce on the other side an enlarged inverted image (first image). If a screen be placed in this position, the image will appear as a picture on it. If no screen be interposed, but another lens (eyepiece), of longer focal length, be placed between the

observer's eye, and this image, at a distance from the first image of a little less than its focal length, the rays from the image in passing through this latter lens are refracted in such a manner that an image of this image (second image) is produced, resulting in further magnification.

Fig. 206 is a sketch of a Leitz microscope, and one that is very suitable for textile purposes.

Before using a microscope the essential parts should be known, and a student using one for the first time should seek the aid of some person who has previously used one, as the instrument can be damaged very easily.

The particular microscope illustrated consists of a brass stand with a substantial base—to the stand being attached, by means of a rack and pinion, a brass tube. Screwed to the bottom of this tube is a set



of lenses called the *objective*; and fitting in the top is another set termed the *eyepiece*. The tube also can be made longer by the manipulation of the *draw-tube*.

Under the objective is a brass platform with a hole in the centre known as the *stage*. On this is placed the glass *slide* containing the object to be examined under the microscope. Beneath the stage is a *mirror* capable of turning in all directions, to reflect the light through the object.

The magnifying power of the microscope is obtained by the combination of objective and eyepiece, and (if necessary) increasing the distance between them by using the draw-tube.

Using a  $\frac{1}{4}$  inch objective and a No. 1 eyepiece, the magnification is about 60 lineal multiplications, i.e. 3,600 times the real area; but magnifications are always expressed as lineal—called diameters.

Using a  $\frac{1}{2}$  inch objective and a No. 3 eyepiece, it is possible to magnify to 450 diameters. This is quite high enough for all ordinary purposes in textile chemistry.

*To Use the Microscope*

First screw in the objective, insert the eyepiece and place the instrument on a very firm table or bench. Arrange the source of light at a suitable distance from the base, and look down the tube with one eye. Turn the mirror until the brightest effect is produced.

Next prepare the slide—instructions for which will be given in the proper place—and place it on the stage so that the ends are held by the clips, and the portion under the cover glass is over the hole in the stage.

Look down the tube and carefully turn the rack and pinion until the object is *nearly* focused, then turn the micrometer screw which gives the fine adjustment, until the object is *exactly* focused.

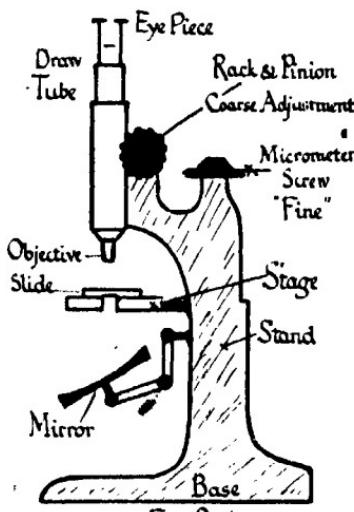


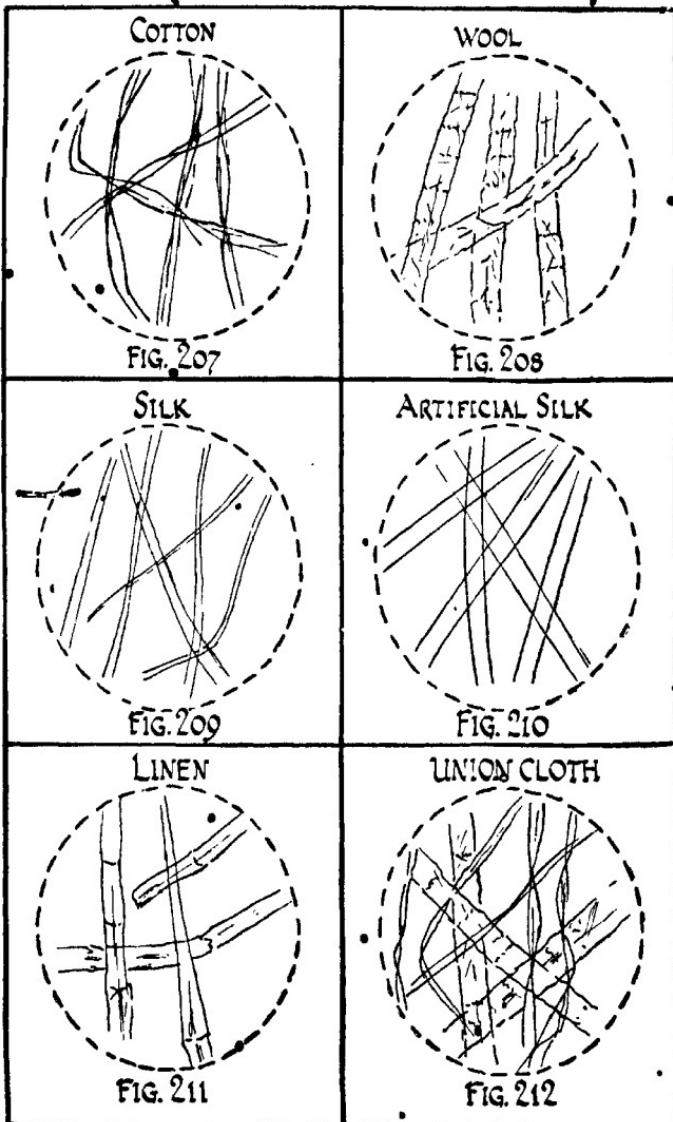
FIG. 206

It requires only practice to learn to correctly manipulate a microscope, and anyone after a few patient trials should be able to use it. Cover glasses should be used always—even over cotton fibres—and it is often an advantage to mount the specimen in liquid.

The microscopic appearance of fibres as represented in textbook diagrams is frequently much more ideal than that usually met with, and the beginner often fails to recognize the specimen unless it has been specially prepared.

The illustrations here given (Figs. 207-212) are drawings from actual photographs of samples as usually met with, and have not been specially selected.

Slides should be made by teasing out fibr's with a needle and placing a few in the middle of the glass. A drop of water, or if the specimen is to be made permanent, a drop of Canada balsam in xylol,



is put on the fibre and another in the centre of a cover-slip, which is then inverted and allowed to fall gently on the tuft of fibres.

A piece of filter paper is put on the top, and gentle pressure is applied to the slip to distribute the liquid and to expel excess—which is absorbed by the paper. The slide is now ready for examination.

The student should first prepare slides of known fibres from several sources, and secondly examine slides made by others, before he attempts to examine the fibres from unknown fabrics.

*Carefully drawn sketches should be made of every fibre examined and, as far as possible, the drawings should be to scale.*

A microscopic accessory known as a camera lucida is a very great aid in this connexion. The form made by Leitz to slip into the top of the tube after removing the eyepiece is a very good one. By using this addition a sheet of paper can be placed at the side of the microscope, which appears superimposed on the image when looking through the eyepiece. The observer has now merely to look down the tube, and by bringing a pencil into the field of view on the paper he can sketch over the image as it appears on the paper.

#### THE ACTION OF HEAT AND VARIOUS REAGENTS ON FIBRES

The effect of heat on fibres should be studied by heating a small tuft in a narrow test tube. The nature of the gas evolved and the appearance of the residue should be carefully noted.

Silk and wool give off ammonia, and a smell of burnt horn or feathers is noticed. Wool sometimes evolves sulphur dioxide after liberating ammonia. Cotton yields very little odour, and usually evolves an acid gas.

*Acetic acid* has no action on cotton, wool, or silk.

*Diluted ammonia 1 : 1* has also no effect on them.

*Caustic potash or soda* (10 per cent.—20 per cent. strength) dissolves wool.

*Stronger caustic alkalis*, while not dissolving cotton, cause it to shrink and become gelatinous on the surface.

*Dilute mineral acids* (nitric, sulphuric, hydrochloric) have very little effect on fibres until they are removed from the liquid and dried, when cotton is converted into hydro-cellulose, which falls to a powder on touching. Wool and silk are not destroyed, but if the acid used is nitric, they acquire a yellow colour which is much intensified if the fibre is afterwards dipped into ammonia.

*Concentrated acids*, as a general rule destroy or dissolve all fibres. Strong sulphuric dissolves silk in the cold, wool on heating, and causes cotton to swell up to a gelatinous mass which is soluble in water.

*Bleaching powder*, if properly used, does not attack cotton, but

wool and silk fibres are both destroyed by it, if the action is continued for some time.

*Chlorides of magnesium, zinc and aluminium* when dried on the fibre liberate hydrochloric acid, which, acting on cotton, destroys it by converting it into hydro-cellulose. This action is sometimes used to determine approximately the proportion of wool in a union cloth.

#### CHEMICAL TESTS FOR THE IDENTIFICATION OF FIBRES

Many reagents have been suggested for the testing of fibres. Detection when fibres of one class are not mixed with those of another is, as a rule, not very difficult, and the following scheme is sufficient for most cases:—

1. Heat in an ignition tube—note odour and nature of residue.
2. Immerse in dilute hydrochloric acid, and when completely saturated, remove and dry on an asbestos mat over a small flame without charring.
3. Boil for a few minutes in 10 per cent. caustic soda solution.
4. Treat with cold concentrated sulphuric acid.

#### PROPERTIES OF THE COTTON FIBRE

In the natural condition cotton fibre (raw cotton) as received at Liverpool contains about

88–89 per cent. cellulose (including ash),  
 7–8 per cent. moisture,  
 1 per cent. natural wax, etc.,  
 2–3 per cent. foreign impurities.

Most of these substances can be separated from the cellulose by suitable treatment, e.g.:—

1. Boiling in very dilute (1 per cent.) caustic soda solution.
2. Thorough washing in water.
3. Steeping in strong *cold* hydrochloric acid.
4. Very thorough washing until all trace of acid is removed.
5. Drying in a steam oven.

The following results were obtained by so treating a sample of American cotton straight from the bale on arrival at the Blackburn Technical College:—

Moisture = 7·1 per cent. Cellulose = 89·2 per cent. Impurities removed = 3·7 per cent.

An interesting series of experiments has been conducted in my laboratory to determine what influence the various processes through which the fibre passes, to make it into yarn, have on the removal of or addition to these impurities.

The same bale was sampled at various stages, and the samples

carefully treated with the same reagents under the ~~same~~ conditions.  
The following results were obtained :—

**Scutched cotton after cleaning and opening—**

Moisture	.	.	.	.	.	.	4.3 per cent.
Cellulose	.	.	.	.	.	.	90.0 per cent.
Impurities	.	.	.	.	.	.	5.7 per cent.

**Carded cotton—**

Moisture	.	.	.	.	.	.	4.3 per cent.
Cellulose	.	.	.	.	.	.	90.0 per cent.
Impurities	.	.	.	.	.	.	5.7 per cent.

**Drawn sliver—**

Moisture	.	.	.	.	.	.	4.3 per cent.
Cellulose	.	.	.	.	.	.	90.0 per cent.
Impurities	.	.	.	.	.	.	5.7 per cent.

**Roving—**

Moisture	.	.	.	.	.	.	5.4 per cent.
Cellulose	.	.	.	.	.	.	90.0 per cent.
Impurities	.	.	.	.	.	.	4.6 per cent.

**Mule-spun yarn—**

Moisture	.	.	.	.	.	.	4.3 per cent.
Cellulose	.	.	.	.	.	.	91.35 per cent.
Impurities	.	.	.	.	.	.	2.35 per cent.

After spinning, cotton yarn is "conditioned," i.e. treated with a fine spray of water to give it the moisture necessary for imparting pliability—perfectly dry cotton being brittle. As cotton has hygroscopic properties which enable it to absorb up to 8 per cent. of moisture (on the average) from the atmosphere, cotton yarn which contains that amount of moisture is called "natural cotton." Anything in excess is illegitimate.

Some spinners add calcium chloride or magnesium chloride or both, sometimes with, sometimes without, the addition of zinc chloride, thereby increasing the hygroscopic property of the yarn—which enables water to be sold as cotton.

Yarn should be tested therefore for the presence of "chlorides" by steeping it in warm distilled water for some time and testing the liquor with silver nitrate solution. A white precipitate, insoluble in nitric acid and soluble in ammonia, proves the presence of chlorides.

In some cases it may be desirable, and even necessary, to add zinc chloride when conditioning, e.g. in very coarse yarns which will be woven up without any application of size and where no antiseptic can be added in the usual way to prevent mildew.

A considerable increase in "ash content" indicates presence of metallic impurities or adulterations.

Raw cotton ash is about 1 per cent. on the average.

Samples examined in my laboratory at various stages in the spinning process gave the following results. As before, the samples all came from the same bale of American cotton :—

Raw cotton from bale . . . . .	1·43 per cent.
Scutched cotton . . . . .	1·45 per cent.
Carded cotton . . . . .	1·43 per cent.
Drawn sliver . . . . .	1·43 per cent.
Roving . . . . .	1·43 per cent.
Mule-spun yarn . . . . .	1·43 per cent.

In all cases the percentages are calculated on the *dried* cotton.

The actual methods I adopted in making the analyses of the samples of fibre were :—

*For Moisture.* A light aluminium box about three inches in diameter and nearly an inch deep, with a tight-fitting lid, was dried in a steam oven and then weighed on a balance sensitive to one milligram. The vessel actually used was a case that had contained Gibb's dentifrice.

It was then filled with fibre—30 grams being used. After weighing again it was put into the steam oven for several hours, at the end of which time the lid was put on quickly, and when cold the box was weighed. The drying process was repeated for an hour, and if a further loss was obtained it was reheated until the loss was constant.

*For Ash.* The dried and weighed sample was transferred from the aluminium dish to a weighed small evaporating dish made of silica, and heated over an ordinary bunsen burner (*not* a blowpipe) until all carbonaceous matter was burned away, and then weighed. A cylindrical screen of metal about six inches in diameter was arranged round the dish to prevent draughts carrying away any of the very light particles of ash.

The same method of procedure will be a suitable one for students to follow.

After weighing, the ash can be dissolved in dilute nitric acid and tested for chlorides by silver nitrate, and for zinc, magnesium, and calcium by the reactions given in Section XII, pages 154–157.

#### PROPERTIES OF THE WOOL FIBRE

Natural wool is a much more impure substance than natural cotton, and "wool washing" is an important Yorkshire industry, the waste products from which have been for many years a source of great trouble

to both washers and public bodies responsible for the prevention of pollution of watercourses.

Raw wool may contain :—

In the " unwashed " condition—

30 per cent. to 80 per cent. of dirt and other substances removable by washing.

8 per cent. to 12 per cent. of moisture (in warm weather).

8 per cent. to 30 per cent. of moisture (in damp weather).

The composition of " raw wool " in the *dry* condition is usually quoted as :—

Yolk and suint, 12 per cent. to 47 per cent.

Wool fibre, 72 per cent. to 15 per cent.

Dirt, 3 per cent. to 24 per cent.

Washing with very dilute alkali or soap is capable of producing a wool largely free from grease and filth. Extraction with organic solvents has been tried, but it has not been adopted on the commercial scale, owing to the fact that the reagent acts too keenly and spoils the natural properties of the fibre.

A series of experiments conducted in my laboratory with wool taken straight from animals reared and grazed on the borders of Lancashire and Yorkshire yielded the following results :—

Raw wool as pulled from fleece	=	100 gm.
--------------------------------	---	---------

Raw wool after washing with pure soap, then	=	
---	---	--

water, and exposing to the atmosphere	=	55 ,,
---------------------------------------	---	-------

The same washed wool dried at 100° C. till	=	
--	---	--

moisture was expelled	=	48 ,,
-----------------------	---	-------

Ash obtained from same washed and dried sample	=	0·6 ,,
--	---	--------

Ash obtained from original wool	=	14·5 ,,
---------------------------------	---	---------

" Conditioning " is determined by the loss in weight of a sample dried at 105° C. to 110° C.

The methods used for determining moisture and ash in wool are similar to those used for cotton.

Wool, like cotton, is somewhat hygroscopic. The standard allowed for natural wool by the Bradford Conditioning House is 18½ per cent.

Silk is the fibrous substance spun by the " silk worm " to form its cocoon. It resembles wool in many respects.

There are two classes—reared and wild.

The former comes chiefly from China, Japan, India, Italy, South of France, and Greece.

It is secreted by the grub as two separate liquids which run into a common channel at the exit where it solidifies, thus forming a uniform double layer.

This thread is reeled off from the cocoon by putting it in warm water to soften the gum with which it is surrounded. As a rule from

5 to 20 separate "ends" are collected and reeled off as one thread, which may be anything from 1,000 to 4,000 yards long.

Silk is hygroscopic, particularly in very damp air, and it can absorb nearly one-third of its own weight without *feeling* damp.

The legal limit for moisture is 11 per cent.

It is very elastic and strong and has an average diameter of 0.007 inch.

It is composed of silk gum, silk fibre, water, colouring matter, fatty materials and mineral (ash).

The gum is soluble in hot water or soap solution—it forms nearly one-quarter of the weight of the raw silk.

The fibre when purified is found to contain carbon, hydrogen, oxygen, and nitrogen. It is called fibroine.

• The ash content should not exceed 0.7 per cent. to 1 per cent.

Silk is very readily dissolved by cold concentrated hydrochloric acid, hot caustic alkalis, basic zinc chloride, and ammoniacal nickel oxide solution.

It is capable of absorbing large quantities of metallic compounds, particularly tannate of iron, with which it is often "weighted."

Determination of the ash content will detect this adulteration.

## SECTION XIV

### THE MACHINERY

POWER-DRIVEN textile machinery developed very rapidly during the latter half of the eighteenth century, and this was largely responsible for the removal of the industry from the East, South, and West of England to the North.

The hand loom has now disappeared (except from museums), and the power loom has taken its place. Mules are no longer turned by hand, and sizing, bleaching, dyeing, and printing are all mainly power machine processes.

Now successful "power" machinery necessitates :—

1. Abundant and cheap fuel—such as coal or oil.
2. Abundant and soft water—for steam-raising purposes, washing, bleaching, dyeing, etc.
3. Efficient lubrication—to reduce friction and wear-and-tear, and to increase speed.

A textile manufacturer who neglects to attend to these essentials is giving something away to his competitors.

*Coal* should be examined for ash, moisture, and calorific power; and these determinations can be carried out quite easily in any mill with very simple apparatus.

Before testing, the coal should be carefully sampled so as to get a truly representative specimen. The sample should be ground up in an iron mortar or small grinding mill, mixed by sieving ("60 mesh") and immediately bottled in a well-stoppered vessel.

To DETERMINE MOISTURE. Heat between 50 and 100 grams in a porcelain dish to 100° C. to 105° C. until no further loss occurs.

The amount present should not exceed 8 per cent.

To DETERMINE ASH. About 5 grams should be weighed in a silica basin and carefully heated over a bunsen flame or in a muffle furnace, taking care not to fuse the ash.

Coal from different sources shows great variation in ash content.

The following are some results obtained from standard textbooks on fuel :—

36 samples from Wales	average	4.91 per cent.
18      "      Newcastle	"	3.77      "
28      "      Lancashire	"	4.88      "
8      "      Scotland	"	4.03      "
7      "      Derbyshire	"	2.65      "

15 samples of slack or small coal for stationary boilers :—

Average = 16 per cent. Lowest = 9.3 per cent. Highest = 22.27 per cent.

*Cannel Coal*—large number of samples :—

Highest = 15 per cent. Lowest = 9 per cent. Average = 13 per cent.

\* Results obtained by me in the examination of samples of coal (mostly to be used for firing mill boilers) :—

Highest, 14.2 per cent. Lowest, 4.8 per cent. Average, 12 per cent.

Results obtained in the laboratory of the Blackburn Technical College during the past twelve months :—

No. of samples, 80. Highest, 12.3 per cent. Lowest, 5.2 per cent. Average, 9.8 per cent.

To DETERMINE CALORIFIC VALUE. When coal burns in air or oxygen, or in any other medium capable of sustaining its combustion, the chemical changes thereby produced result in the *liberation of heat*. In determinations of calorific value attempts are made to measure the amount of heat evolved by the complete combustion of 1 lb. of coal.

Quantities of heat can be expressed in several ways, but for calorific values of coal it is most usual to do so in what are known as *British Thermal Units* (written B.T.U.).

One B.T.U. is the amount of heat required to raise one pound of water through one degree Fah. in temperature.

Occasionally it may be necessary to use other standards; in that event, use the following factors :—

To convert B.T.U.s to kilogram calories  $\times .252$ .

To convert B.T.U.s to centigrade heat units  $\times .55$ .

To convert centigrade heat to B.T.U.s  $\times 1.8$ .

Calorific values can be calculated by using a formula, but the results obtained are not so satisfactory as those obtained by direct determinations with a calorimeter.

The standard form of apparatus is the *bomb calorimeter*—an instrument based on that of Berthelot, but it is a very expensive appliance and is not suitable for mill use.

The form used in this college laboratory is the Roland Wild calorimeter, made by Messrs. Alex. Wright & Co., Westminster, S.W., and

is a very reliable and not expensive article. (Cheapest form, about £6 6s.)

Fig. 213 shows the instrument in section. The following description is copied from the makers' pamphlet supplied with the instrument.

The apparatus consists of a combustion chamber A suspended from the cover by conduit C, which is furnished with a valve D.

E is a copper vessel containing water, surrounded by an air chamber F to prevent radiation.

G is a Fah. thermometer graduated in 1/10ths. H is a paddle stirrer. The water value of the instrument is 70 grams. (This has been determined by the makers and varies with the instrument.)

In using this instrument a small quantity of coal is burned in A by mixing it with sodium peroxide, the heat evolved from its combustion being absorbed by the water surrounding it. If the weight of the water be known, and the rise in temperature which it sustains measured,

then the amount of heat evolved by the coal can be calculated, if certain corrections be made.

It has been found, if *exactly* 0.73 gram of coal be mixed with 12 to 15 grams of sodium peroxide and rapidly fired—which is done by heating a small piece of nickel wire and dropping it down the conduit—that the rise in temperature of water in degrees Fah.  $\times 1,000$

= calorific power of 1 lb. of fuel in B.T.U.

The coal must be *dry* when weighed, and should pass through a 60-mesh sieve. An error of 0.01 gram will produce an error of nearly 1.4 per cent. in the result.

The reason for using 0.73 gram instead of 1 gram is that, owing to certain chemical changes occurring that are not produced when coal burns in air, more heat is registered than is actually evolved by the combustion of the coal. The makers have found that this = 27 per cent. of the total heat evolved, and they correct for it by using the smaller quantity of coal in the experiment.

The absorption of heat by the calorimeter itself is corrected for by using 1,000 grams of water less the water value, in this case 1,000 — 70 = 930 grams.

Through the kindness of the Blackburn Electrical Engineer (Mr. Wheelwright), I have been able to check the values obtained by a

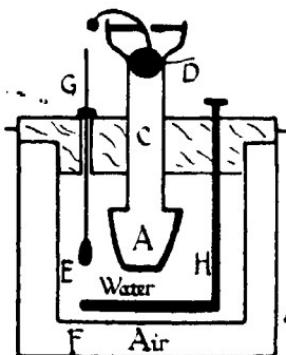


FIG. 213

Roland Wild instrument with those obtained from the same coals by the bomb calorimeter used at the electricity works.

Sample No.	Calorific Power in B.T.U.s.	
	By Roland Wild.	By Mahler Bomb.
1	10,750	10,800
2	12,400	12,450
3	12,150	12,000
4	12,800	12,750
5	12,500	12,600
6	12,700	12,730
7	10,900	11,000

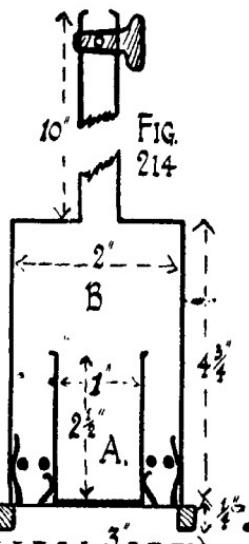
A much older, but still very good form of calorimeter is the Lewis Thompson, which can be made by any metal worker. A dimensioned diagram of the instrument is given in Fig. 214.

The combustion chamber A is held to the base by means of a spring. The cover B is attached in a similar manner to the same base. It is provided with a brass tube closed near the top with a tap, and holes are bored near the bottom.

Coal is mixed with an oxidizing substance, put into A with a fuse, the end of which is ignited. The cover B is put on quickly, the tap shut, and the instrument lowered into a vessel containing a known quantity of water at a known temperature. In a few seconds the burning fuse ignites the mixture and the hot gases produced escape through the holes in the cover, and passing up the water cause a rise in temperature. When combustion is complete the tap is opened and water then rises up inside the calorimeter, thereby absorbing its heat.

The oxidizing mixture consists of 3 parts of potassium chlorate, 1 part of potassium nitrate, thoroughly dried and perfectly mixed by hand on a piece of paper, *not in a mortar*.

For each charge 2 grams of dried coal is mixed with 20 grams of oxidizing mixture.



The fuse is made by soaking cotton wick in a solution of potassium nitrate and drying in a steam oven.

It is desirable to use about 2 litres of water in the outer vessel, which should be tall and not very wide, to insure the thorough cooling of the escaping gases.

Then, if no correction is made for heat lost by radiation and absorption by the instrument, calorific power of coal in B.T.U. =

$$\frac{\text{Rise in temperature in } {}^{\circ}\text{F.} \times \text{weight of water in grams}}{\text{Weight in grams of coal used}}$$

As a rule an allowance of 10 per cent. is made to correct for heat absorbed by the apparatus, decomposition of substances in the oxidizing mixture, and solution of substances remaining. Loss due to radiation is compensated for by commencing the experiment with the temperature of the water a few degrees below the temperature of the room.

Corrected formula then is :—

$$\text{C.P. (in B.T.U.)} = \frac{(\text{rise in temperature}) - (1.10 \text{ rise}) \times 2,000}{2}$$

**THE EXAMINATION OF FLUE GASES.** It is not sufficient for a mill-owner to know how much heat his fuel is capable of evolving. He should know also how efficiently it is being used. In this connexion it is most desirable systematically to test the gases escaping through the flues and up the chimney stack.

If complete combustion of the fuel has been effected, the flue gas should contain nitrogen and carbon dioxide only; as a matter of fact oxygen and carbon monoxide are present also.

A simple but very efficient apparatus designed by the author for the analysis of flue gases is shown in Fig. 215. The graduated glass vessel A is taken to the flue and filled by suction or other convenient means and the screw clips on the pieces of rubber on the ends tightly closed. It is then brought to the laboratory or testing room and attached to the funnel as shown in Fig. 215.

Water is run into the funnel B, the rubber tubing squeezed to remove any air bubbles, and the clip at the bottom carefully opened. The funnel is moved up or down until the water stands at the same level in each vessel, and the volume of gas in the graduated vessel is noted.

The clip is closed, the funnel emptied of water and caustic soda solution put in. If the level of liquid in the funnel is kept high, some of it will be forced into the graduated vessel, when it will gradually absorb the carbon dioxide. The rate of absorption may be increased by first closing the clip, and then turning the vessel on its side. More soda solution is put into B as the liquid passes into A during absorption.

When absorption appears to be complete, the levels should be adjusted again, and the volume of gas now in the vessel determined.

To estimate the volume of oxygen, the top of the graduated vessel should be attached to a similar vessel C (Fig. 216) which is quite full of pyro soda solution. When both clips are open and the funnel D attached to the vessel C lowered, or that attached to the vessel A raised, the gas can be passed completely from one to the other (A to C). The clip E is then closed.

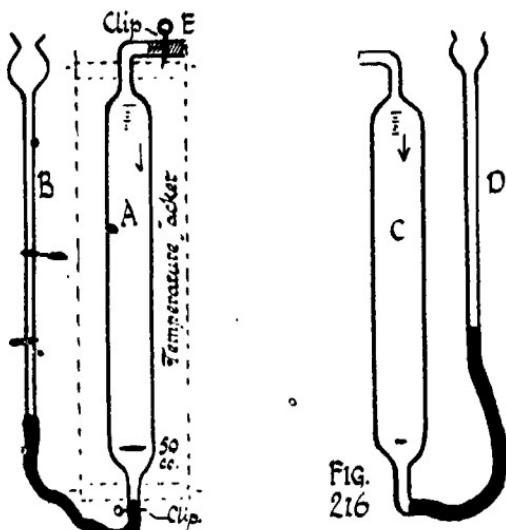


FIG. 215

FIG.  
216

After the absorption of the oxygen is complete in C the levels in C and D are adjusted and its volume thus determined.

To absorb the carbon monoxide the pyro soda vessel A is detached, and a similar vessel containing a solution of cuprous chloride in hydrochloric acid is attached to C, and the process of absorption repeated by passing the gas from C into the new vessel.

It may be assumed that the residual gas is nitrogen. The experiment may be performed without "jacketing" the vessels if the temperature of the room in which the tests are being carried out is constant. Should it vary, it is desirable to arrange a water bath as shown by dotted lines in the diagram (Fig. 215).

In analyses of certain flue gases from mills in this district, the following results were obtained. They illustrate the great variations that may be found.

Sample from	Per cent. Carbon Dioxide.	Per cent. Oxygen.	Per cent. Carbon Monoxide.	Per cent. Nitrogen.
No. 4 Boiler (average of 10 expts.) . .	9.93	7.5	nil	82.57
No. 5 Boiler (average of 9 expts.) . .	10.7	6.3	nil	83.0
Economizers :-				
End near boiler . . . . .	6.4	13.4	1.0	79.2
End away from boiler . . . . .	4.9	14.0	0.6	80.5
Another mill-boiler flue . . . . .	10.2	7.5	1.3	81.0

*Boiler feed water should be tested for—*

- (a) Total solids.
- (b) Total hardness.
- (c) Temporary and permanent hardness.

*Condensed water should be examined at intervals for—*

- (a) Reaction to lacmoid.
- (b) Chlorides.
- (c) Iron.

*Softened water should be examined at regular and frequent intervals for—*

- (a) Total solids.
- (b) Hardness.

The instructions for carrying out these tests will be found in Section V, pages 43-44.

Iron is best estimated by a colorimetric process using either potassium sulphocyanide (thiocyanate) or, in the absence of lead, hydrogen sulphide.

A measured volume of water is put into a Nessler jar and a little of the reagent added to it. Similar jars are filled with the same quantities of distilled water to which have been added known amounts of iron solution. The liquids which give the same tint of colour on addition of the same reagent are assumed to contain the same amounts of iron.

*Boiler compositions* are used to precipitate the dissolved solids of the boiler-feed water in a friable and easily removable condition. Many different substances are used, but the most popular, and probably the most efficient, are mixtures of caustic soda and soda ash.

Proportions that are suitable for one feed water may not be desirable for another, and it would often pay the mill-owner to seek advice regarding the best combination to use.

The following are figures obtained by the analyses of three typical boiler compositions of this class :—

No. 1. Soda ash, 80 lb. Caustic soda, 200 lb. Water, 72 gallons.

No. 2. Sodium carbonate, 52 lb. Caustic soda, 170 lb. Water, 75 gallons.

No. 3. Sodium carbonate, 42½ lb. Caustic soda, 89 lb. Water, 90 gallons.

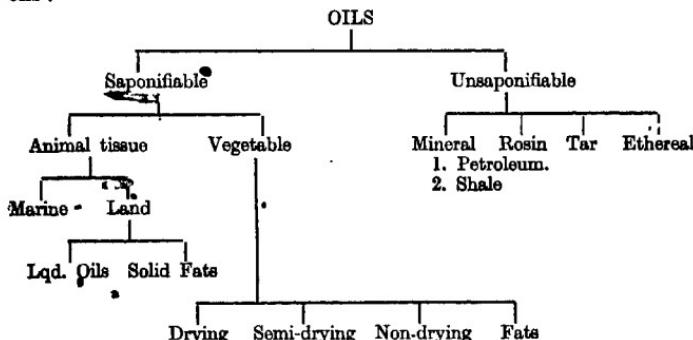
Oils form a very important class of mill stores, and all of them should be carefully scrutinized before use.

They are applied in textiles for :—

- (a) Lubrication—e.g. looms, and cylinders of engines.
- (b) Softening wool.
- (c) Giving pliability to cotton yarn.
- (d) Certain mordanting and finishing processes.

Oils have been extracted from animal and vegetable tissues from the earliest times, but during the last hundred years they have been obtained also from mineral sources—but these *mineral* oils are of a composition quite different from that of the fatty oils.

The following table illustrates the usual method of classifying oils :—



Oils are all (with the exception of castor oil) very sparingly soluble in cold alcohol.

Animal oils are usually extracted by rendering (heating to burst the tissue).

Vegetable oils are largely “expressed,” although extraction with solvents is becoming more important every day.

These processes may be illustrated on a laboratory scale by the following experiments :—

*Rendering.* Take a large and deep evaporating dish, half fill with water, add pieces of cocoa-nut kernels and boil up. When the oil is extracted, cool and skim off the thin layer of solid fat, remelt, cool and dry on filter paper.

*Expressing.* Make a small press similar to that shown in section in Fig. 217. Grind some good-quality linseed in a grinding mill or coffee-grinder, and put it in the cavity and screw down the press. Collect the oil as it runs out. If it is not quite clear, filter it through a *dry* filter paper.

*Solution.* Expression always leaves a certain amount of oil behind in the cake—from 5 per cent. to 10 per cent. The cake from the previous experiment can therefore be used for this experiment. Fit up the apparatus shown in Fig. 218. Put the solvent, which may be carbon disulphide, benzene, petrol, or ether in the flask, and the cake—after wrapping in filter paper—into the extraction tube. If ether or carbon disulphide be used the flask should be heated on a water bath as shown. The stream of water through the condenser should be regulated so that all vapour escaping from the extraction tube is condensed. The solvent passes from

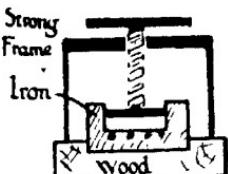


FIG. 217

the tube to the flask automatically when the level of the liquid reaches the top of the siphon tube. After collection in the flask, the solvent is evaporated off and the oil remains behind.

The vegetable and animal, or fatty, oils used for textile purposes include palm, cocoa-nut, rape, soya bean, linseed, castor, cotton-seed, olive, whale, and sperm.

Specimens of all these oils should be examined and some of their constants determined.

*Mineral oils* are hydrocarbons (Section X, pages 114–117). They are not acted upon by caustic alkalis, to produce soaps, and are on that account said to be *unsaponifiable*.

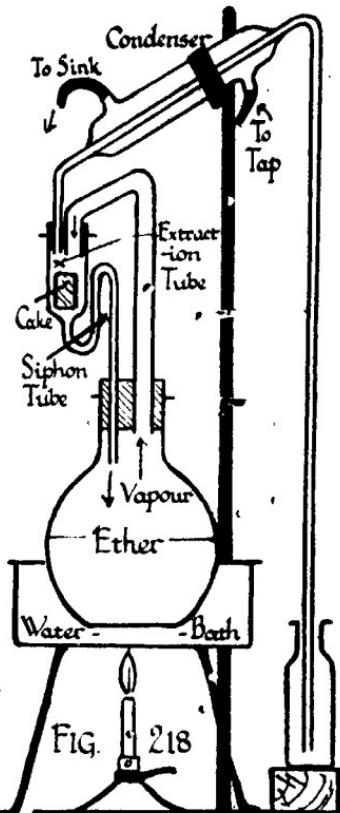


FIG. 218

Rock-oils, as they are sometimes termed, are now obtained from many parts of the world, and the industry has attained enormous dimensions, although it was only in 1859 that the first Pennsylvanian oil well was drilled by the modern method of "tubing." These modern wells vary in depth, some being 5,000 or 6,000 feet deep.

The crude oil is fractionally distilled under reduced pressure, the different fractions being sold for various trade purposes under different names.

In Scotland an oil-bearing shale is treated in a similar way, to obtain the *Scotch shale oils*, an industry which commenced in 1847.

The chief fractions prepared from petroleum oil are :—

Motor spirit or benzine (at 30° C.-140° C.)	sp. gr.	·650-·720
Solvent naphthas . . . . .	"	·700-·740
Illuminating oils . . . . .	"	·790-·825
Non-viscous spindle oils . . . . .	"	·850-·870
Viscous machinery oils . . . . .	"	·880-·920
Viscous steam cylinder oils . . . . .	"	·885-·920

After fractionating, the oils are "refined" by agitation, first with sulphuric acid of sp. gr. 1.76 and then with caustic soda (1-2 per cent. solution).

Occasionally oils are found in which the acid has not been completely removed, and sometimes one is met with from which the alkali has not been washed properly. These are serious blemishes in lubricating oils.

*The examination of oils* should include the following determinations :—

1. Very accurate determination of *specific gravity*. This should be done at 15.5° C. or 60° Fah. in a specific-gravity bottle (Section III, page 29). Sometimes only a very small quantity of oil is available, not sufficient for the usual methods. In that event mixtures of alcohol and water should be made until small drops of oil will become perfectly spherical in one of them, and exhibit no tendency to rise or sink. The density of this liquid mixture is then equal to that of the oil, and can be used in lieu of the oil for filling the specific-gravity bottle.

The following results were obtained by *students* working in our laboratories with *bona fide* trade samples of oils sold for textile purposes :—

Rape = 0.913. Camphor = 0.964. Olive = 0.92. Loom = 0.903.  
 "Stainless" Spindle = 0.91. Neat's Foot = 0.91. Cylinder = 0.92.  
 Sperm = 0.88.

2. Determination of *Ash*. The method of doing this has been given in Section III, pages 32-33.

As the ash of oils is very small, and really should be nil, a large crucible, or a small silica dish sufficient to hold about 10 grams of oil, should be used.

3. Presence of *acids or alkalis*. Shake some up with warm water, and test the water with—

- (a) Methyl orange—turned pink by mineral acids.
- (b) Phenolphthalein tincture made faintly pink with one drop of alkali—decolorized by fatty and mineral acids.
- (c) Phenol phthalein tincture—turned pink by alkalis.

4. Determination of *flash-point*. This is the temperature at which the vapour evolved from a sample of warm oil will ignite at the surface of the liquid when a small flame is put near it.

There are two variations of the method of performing the experiment—the “open” test and the “closed” test. In the former, the vessel in which the oil is heated is without lid, and in the latter a lid is used in which is a small hole that is kept closed until the temperature has nearly reached that at which vapour is evolved. Then it is opened for an instant and a small flame applied at the opening. If no flash results, it is closed and the process repeated at higher temperatures.

Standard forms of apparatus are available for performing these tests, but as a matter of fact they are not really necessary to get a result sufficiently accurate for ordinary purposes.

The method adopted in our laboratories is to sink a large crucible in sand in an iron tray and fill it to within  $\frac{1}{2}$  inch of the top with oil.

If the “closed test” is being performed, a small tin lid is put on the top. A small hole has been bored in this lid and is closed by putting over it a piece of broken electric light carbon (Fig. 219). A small flame about the size of a pea is obtained by connecting a mouth blowpipe to the gas supply. The thermometer must dip well into the oil. When the test is made the gas carbon rod is removed for a second with one hand and the flame applied to the hole with the other. If the flash-point has been reached a pale blue flame flashes along the surface under the lid. The temperature indicated by the thermometer is then read, which is the flash-point.

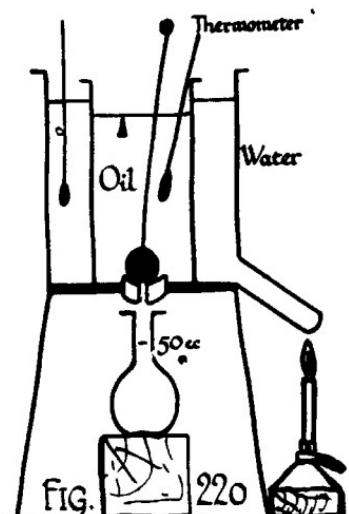
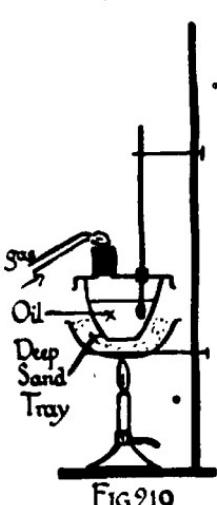
In the “open test” no lid is used and the flame should be brought near the surface.

The “open flash-point” is higher than the “closed” one for the same substance. If the experiment is repeated, a fresh sample of oil must be used, as some of the volatile products are lost to it by the first heating.

Some results obtained in our laboratory by first-year students :—

Description of Sample.	Open Test.		Closed Test.	
	° C.	° Fah.	° C.	° Fah.
1. Motor gear oil . . . . .	256	493	242	408
2. Cylinder oil . . . . .	217	423	207	405
3. Spindle oil. . . . .	200	392	175	347
4. " "	197	387	179	354
5. Engine oil . . . . .	220	428	198	388
6. Spindle oil. . . . .	155	311	145	293

5. *Viscosity* determinations. By viscosity is meant the internal friction exhibited by liquids. Those possessing the minimum of viscosity are said to be mobile. No satisfactory laboratory method has yet been devised for measuring viscosity *directly*. The usual method is to measure the rate of flow of the liquid through a small orifice. The instrument used is called a viscometer, and the favourite form in use in this country is that invented many years ago by Redwood and still called by his name.



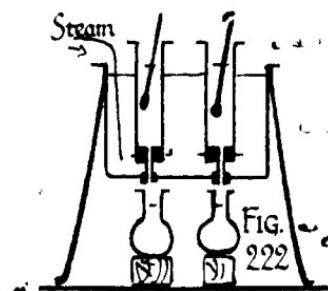
His instrument consists of a metal vessel to contain the oil, the bottom being provided with a small orifice made by boring a hole through an agate cup. This vessel is surrounded by another containing water that can be heated by means of a side tube (Fig. 220). The hole in the agate is closed by a metal ball, which when raised allows the oil to run out into a 50 c.c. flask placed underneath.

The time of flow is recorded and compared with that taken by 50 c.c. of pure rape oil. A further precaution is necessary if the two results are to be comparable, namely the heights of the columns of oil must be identical. This is provided for by placing a pointer near the top of the oil vessel, to the apex of which the level of the liquid is adjusted in each case.

Redwood found that the average time taken by 50 c.c. of rape oil at 60° Fah. was 535 seconds. This he called 100 on his scale, and he also suggested a correction for sp. gr.

Viscosity on Redwood scale, using a Redwood viscometer :—

$$\frac{100 \times \text{time of flow} \times \text{sp. gr. of oil at temperature of flow}}{535 \times .915}$$



For approximate work comparisons of viscosities may be made by selecting a 25 c.c. pipette, making a mark on the lower stem, and then finding the time taken by the oil to run between these two points when the pipette is held in a vertical position.

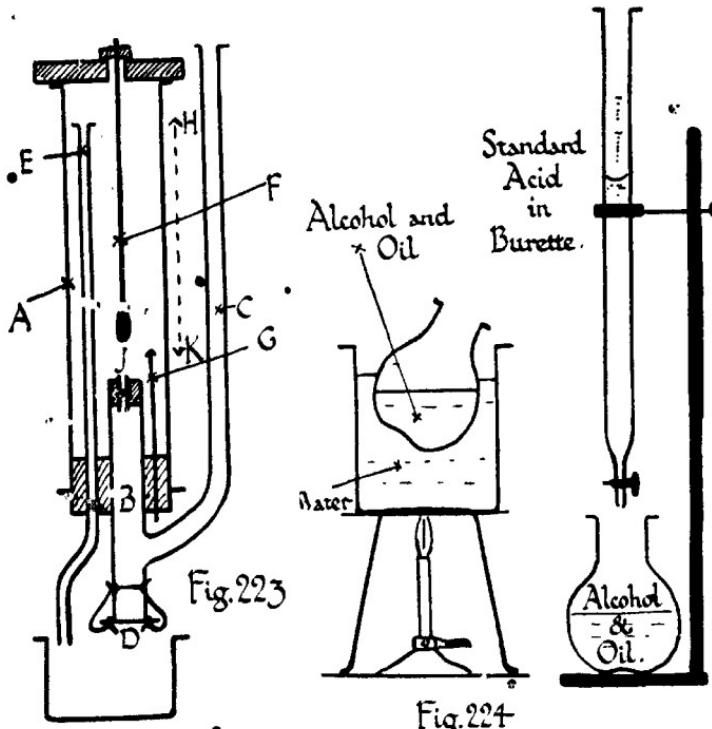
Another simple form is shown in Fig. 221, made from a piece of glass tubing half an inch in diameter, the end being provided with a rubber bung through which passes a small piece of capillary tubing.

Fig. 222 shows the same form of apparatus arranged in duplicate, so that comparisons can be made at temperatures differing from that of the room. The tubes containing the oil and the efflux capillaries must be the same diameter in each case.

Fig. 223 is a form recently designed by the author for use in his laboratory, from which very satisfactory results have been obtained.

This new form of viscometer consists of an outer glass vessel A, in which is a glass tube B, carrying the efflux tube J, and which is closed by means of a clip D.

The oil is passed into the vessel by means of side tube C. When it reaches to the top of tube E, the surplus fluid is carried off through it.



The thermometer F is so arranged that the bulb is near the efflux tube J.

When carrying out the experiment the clip D is opened, and at the same instant the time is noted. The oil is allowed to flow until the surface just reaches the point K of the thin glass rod G, and the time again noted.

If, when cleaning the instrument, the positions (in the rubber stopper) of tubes E and B and rod G are unaltered, the head (K—H) and volume of liquid used are identical for each experiment, and thus the relative times of flow will give correct figures for relative viscosities.

The instrument has given very consistent results and has distinct advantages over the Redwood viscometer in certain particulars.

Viscosities at higher temperatures than normal are made when the instrument is surrounded with a hot-water jacket, which is preferably heated by means of "live steam."

Some results obtained with this viscometer are compared in the following table with those obtained at the same time with the same oil using a Standard Redwood pattern.

Description of Sample.	Temp.	Time in Seconds.		Ratio C/B.
		Redwood's.	Cooper's.	
Rape oil . . . . .	62° F.	415	527	1.27
Sperm oil . . . . .	63° F.	188	239	1.27
Lubricating oil . . . . .	60° F.	183	232	1.27
" " " . . . . .	140° F.	58	73	1.26
Spindle oil (mineral). . . . .	65° F.	100	126	1.26
" " " . . . . .	100° F.	55	70	1.27
" " " . . . . .	140° F.	40	50	1.26
Machinery oil (mixed) . . . . .	65° F.	330	416	1.26
" " " . . . . .	100° F.	120	151	1.26
" " " . . . . .	140° F.	63	80	1.27

Fatty oils and mixed oils are very liable to contain free fatty acid, and this should always be estimated to determine the degree of rancidity of the sample.

The apparatus used for this purpose is shown in Fig. 224.

About 10 grams of the oil are weighed into a small flask; 25 to 50 c.c. of neutral alcohol are added and the flask and its contents are gently warmed in a water bath.

Standard alkali is put in the burette—usually N/10 caustic soda is used—and after the addition of 2 or 3 drops of phenolphthalein to the flask, alkali is carefully run in until a pink colour is obtained which remains for a few seconds on stirring.

The strength of the alkali being known in terms of fatty acid, the result may be calculated.

As a rule acidity is returned as oleic acid, and 1 c.c. N/10 alkali = 0.0282 grams of oleic.

It is also very desirable to know the percentage of saponifiable oil present if the analysis is to be a complete one, but this exercise is hardly suitable for an elementary worker to carry out, and it is therefore deferred.

## SECTION XV

### SIZING OF COTTON YARN

**M**OST textile students are aware that sizing commenced originally in the early days of cotton manufacture in India, but of the actual date when it was first found desirable to pass cotton threads through rice water in order to assist in the weaving of the fabric, there is no record.

Dating from that period constant additions to, and many improvements in, the important process of sizing have been made.

"Sizing began in necessity, but has ended in something like dishonesty," says a writer on the subject, but he is careful to acquit the manufacturer and sizer of the blame for this state of affairs.

It is hardly possible to hope that we shall ever reach the point of absolutely perfect sizing, but science has done so much for all branches of industry in the past that it is only reasonable to suppose that it will be able to do more in the future, and that in this advance sizing will share.

Now the chemistry of sizing—as trades go—is simple. Sizing was, and still is, largely empirical; many wonderful mixings have been tried and even patented, which in many cases have proved to be more or less useless.

It is only lately that the chemist has been called in to sort out the wheat from the chaff, and to explain the action of the successful materials.

The chemist in his classification of things (see Section IV, pages 34-35) makes three groups: (1) Elements, (2) Compounds, (3) Mixtures. Of these, class 1 comprises the least in number and the members, as a rule, are the easiest to identify. The second is a very numerous class, and its members possess the peculiarity of being perfectly definite in composition, and consequently they answer to certain well-known "tests" (see Section XII, pages 152-157).

The third class is by far the most numerous; it includes nearly all common things, and its members are the most difficult to identify. There is no fixity or certainty about them—they are constantly altering in minor or in important respects.

Now most sizing materials come under the chemical division of mixtures, e.g. flour and all natural grains, tallow, soap, clay, etc. And so when we say that the chemistry of sizing is fairly simple, we must qualify that statement by adding that the application thereof to sizing ingredients is intricate. There is need therefore for the utmost care and accuracy in testing.

As a general rule cotton warps are sized, and weft is used in an unsized condition. Cotton can be sized in the hank, the ball warp, and the tape condition. The reader is referred to books on sizing for detailed accounts of these methods. Generally speaking, the size is a starch paste to which has been added softening, and sometimes weighting, materials.

The ingredients used for size preparation are usually classified as follows :—

1. Adhesives—starches, gums, etc.
2. Softeners—tallow, fats, waxes, soaps.
3. Weighting materials—clay, metallic chlorides.
4. Antiseptics—zinc chloride, salicylic acid, etc.

Some ingredients fall into more than one class, e.g. glycerine and zinc chloride.

### FLOUR

The name flour has no definite significance, it merely means the powdered solid obtained from a grain of a starchy nature, and although in England we restrict the term to mean the inside of wheat, we do not thereby give a much better indication of what it is.

The substances present in wheat flour are found to be considerably dependent upon several very important factors :—

1. The kind of grain used as seed.
2. The conditions under which it is grown.
3. The locality in which it is raised.
4. The method of milling.

And last, but not least, the dealer through whose hands it passes. In representative samples the contents have been found to be :—  
**Starch**, which may vary between 60 per cent. and 70 per cent.  
**Gluten**, which may vary between 2 per cent. and 15 per cent. (occasionally up to 20 per cent.).

**Moisture** is about 13 per cent.; although the conditions of storage can alter this figure considerably.

**Ash** should not exceed 0·9 per cent., and it may be as low as 0·4 per cent.

A good average is 0·5 per cent. to 0·6 per cent.

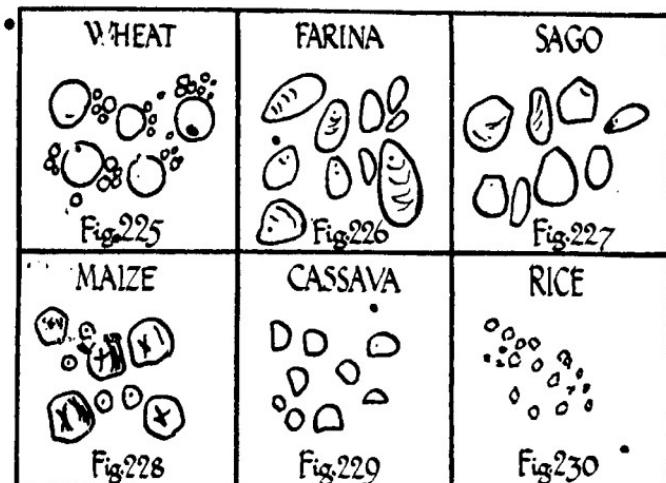
Flour also contains sugar, dextrine, albumen, etc.

That flour contains starch is easily proved by the iodine test. Boil

a little flour and water and cool it by pouring into a jar of cold water. Add two or three drops of tincture of iodine, and a deep blue coloration is produced.

*Note.*—This colour is destroyed by heating or by the addition of caustic soda. Therefore the emulsion must be acid or neutral and cold before the iodine is added. Alkalinity should be neutralized by the addition of acetic acid.

The correct estimation of the amount of starch in flour is too long and difficult for beginners to attempt, but an approximate method is to knead 10 grams of flour in a muslin bag, held in a beaker of water, until all the starch is washed out; allow the starch to settle, pour off the clear water, dry and weigh the sediment.



Starch is a naturally produced body, and hence it has a *structure*. Under the microscope this is very apparent and is found to be *granular*. Again, starches produced by different varieties of vegetable tissues are found to exhibit different granular structures.

This forms a ready means of identification. Figs. 225-230 illustrate the microscopic appearance of the most important sizing starches.

Boiling in water, or the use of certain chemicals, destroys this structure by destroying the outer coating of the granule, which is considered to be a form of starch cellulose, and which does *not* produce a blue colour with iodine. Starch has no adhesive qualities until this coating is destroyed, hence the need for boiling or other treatment to make the starch paste.

Flour is used ~~in size~~ chiefly as an adhesive, and besides starch it

contains another constituent which is still more adhesive. This is termed *gluten*, and the value of flour for sizing purposes is largely determined by the quantity and quality of the gluten present. A good average is 9 per cent. to 10 per cent.

As a great deal of gluten is often lost in fermentation (see pages 200-201), the quality is of more importance than the quantity. This must be tested when moist after washing away the starch. It should be tough, tenacious, and elastic.

A simple and often valuable test to apply to a specimen, to indicate whether the powder is starch or flour, is to add two or three drops of strong nitric acid to some of it placed on a white porcelain lid.

Flour is turned yellow, starch becomes a greyish white translucent mass.

Although flour is not so largely used as formerly, it is still a very important sizing ingredient and is nearly always used (alone or in conjunction with other starches), when it is desired to add weight to the yarn. Before use it must be prepared, which is done by fermenting it after mixing with an equal quantity of water, or by steeping in a solution of zinc chloride.

Fermentation may extend over months, but steeping is considered sufficient if it has lasted two or three weeks. This treatment separates the granules, destroys the stickiness, and dissolves the glutinous products.

#### FARINA

This term is a trade name for potato starch. A potato contains on the average 75 per cent. water, 20 per cent. starch, and 5 per cent. other substances. If potatoes are pulped and washed, the starch can be obtained from them in an almost pure condition.

It is dried carefully at a low temperature, so that it retains about 17 per cent. to 20 per cent. of water.

Farina is characterized by its glistening appearance, its crisp feel, its large granules, and the thick paste it forms with water when gelatinized.

This starch should always be examined under the microscope; the more uniform the granules the better the quality as a rule. Very large granules are not desirable, and a large number of very small ones indicates that the potatoes from which it was manufactured have not matured. Pastes made from these farinas soon lose their adhesive-ness and "fall away."

Even the best farina is liable to exhibit this defect of rapidly deteriorating after making up for size. It can be prevented somewhat by adding a very small amount of caustic soda to the water before the starch is boiled in it.

The ash from farina should be so small as to be almost unweighable if 20 grams or so are completely ignited.

### SAGO

Sago flour is very largely used in North-east Lancashire. In spite of its somewhat yellow natural appearance it is greatly esteemed as a sizing starch, because it greatly strengthens the yarn and allows it to stand a "high pick."

Sago is a starch obtained from the pith of a plant of the palm family, and was first used—according to a Patent Specification—in 1860.

The pith is pulped and stirred in water over a sieve. The starch is thus washed out and passes through the holes while the fibre, etc., is retained. It is then air-dried and shipped.

On reaching this country it is "dressed" by means of silk sieves.

It is usually a nearly pure starch, but the ash is higher than with farina, and is sometimes of a gritty nature.

It presents a very typical appearance under the microscope (Fig 227, page 185). The ends of the granules are distinctly truncated. If too much sago is used in size mixings, the warps are made too stiff, which, if not apparent to the touch, makes its presence evident by cutting the healds.

On this account it is usual in the Nelson district to steep the sago overnight in cold water and boil up in the morning. The boiling should be more prolonged than with farina.

Low-grade sago flour is liable to be contaminated with sea water. It is therefore necessary to test samples for the presence of chlorides.

### MAIZE OR CORN STARCH

This starch is produced in enormous quantities in America, where methods for its extraction have been brought to a high state of perfection. Many years ago there was considerable prejudice against this starch as a sizing ingredient, but to-day it is much more popular, particularly the better brands.

The paste produced from it is very thick, opaque, and somewhat liable to mildew rapidly. When dried it has a harsher feel than that of most other starches, but it is very adhesive and may be boiled for a long time with "open" steam without fear of deterioration. This boiling tends to reduce its natural harshness.

Maize starch is often used in conjunction with flour for heavy sizing.

During the period of the war, when farina was almost unobtainable, it became necessary to use it in "pure" and "light" sizing, and in many cases it is still being retained—so satisfactory has it proved.

Corn starch should be carefully scrutinized under the microscope,

and a sample showing small and regular granules will be found as a rule to give the better results. The ash should be practically nothing.

Many proprietary brands of starchy materials contain maize starch as one of the ingredients.

### CASSAVA

This is a "root" starch produced largely in South America. It is prepared as a food starch under the name of tapioca. It is seldom that the cotton manufacturer buys it in the pure form, although he certainly gets it in certain sizing starches that are sold under special trade names.

Cassava under the microscope appears in somewhat hemispherical granules (Fig. 229, page 185). They gelatinize readily to produce a thin and not particularly adhesive paste.

### RICE

Rice starch is chiefly used as a laundry starch. The qualities which make it desirable for this application are those which militate against its use as a sizing starch.

The granules are small, harsh, and when dried produce a very rough yarn and a cloth of "boardy feel."

Many sizing flours contain small proportions of rice flour or rice starch, these admixtures enabling the user to obtain various cloth effects, particularly in regard to feel.

### LABORATORY EXERCISES IN THE EXAMINATION OF STARCHES

1. Test solubility in cold water by shaking, filtering, and testing the filtrate with tincture of iodine.

2. Put a few drops of a cold emulsion in boiling water. Allow it to cool—note if a jelly is produced. Take some of this in another tube, shake up with more cold water, add iodine, and note production of the blue colour. Boil some of this and note that the colour disappears and probably returns on cooling.

3. To some starch paste add a few drops of caustic soda solution. Test a portion of this with iodine—no blue colour is produced. Neutralize another portion with acetic acid and then add iodine—the blue colour appears.

Therefore, to test for starch always proceed as follows:

Shake up the substance in cold water, boil the mixture, test with litmus paper to determine if it is alkaline. If so, neutralize with acetic acid, cool the liquid, and then add a drop or two of tincture of iodine. If it turns blue, starch is present.

4. Determine the percentage of ash and water in starches. (See Section III, pages 31-33.)

Examine permanent slides of various starches under the microscope and try to identify them. Carefully sketch in your notebook the shape of the granules of each variety.

*Prepare samples of starch for examination under the microscope :—*

Add about enough starch to cover a sixpence to a test tube half full of cold water; shake well and take out *one drop* on the end of a glass rod. Put this on the middle of a clean microscope slide and carefully drop on it a glass cover slip so that no air bubbles are held under it. Place on top a piece of filter paper and gently press.

Prepare in this way samples of farina, flour, maize, and sago; and also farina which has been boiled.

*Flour should be tested for mineral impurities, other starches, and mildew.*

- An increased ash content indicates mineral adulteration, which may also be detected by shaking up the flour in a test tube with chloroform, when the clay, gypsum, etc., settles to the bottom, while flour floats.

Millets, rice, tapioca may be readily detected under the microscope.

Another valuable test to apply to flour is to add to 20 grams of flour a mixture of 70 c.c. of absolute alcohol, 25 c.c. of water, and 5 c.c. of strong hydrochloric acid. Put it in a large tube and digest in a beaker of hot water for some time. Allow it to stand to cool for an hour.

Examine the appearance at the end of that time. If the liquor is—

- (a) *Perfectly colourless*, it is pure wheat flour;
- (b) *Blood red*, it contains ergot;
- (c) *Purple red*, it contains mildew;
- (d) *Yellow*, it contains barley or oat flour;
- (e) *Orange yellow*, it contains pea flour.

*To determine the amount of gluten in flour :—*

Weigh out 20 grams of flour, put it in a 4–5 inch evaporating basin and add water a few drops at a time, stirring with a glass rod until it is made into a lump of dough, *not* a paste. With a little practice, and if too much water is not added, it is possible to gather every particle of the flour into one ball on the end of the glass rod.

Take a piece of washed cotton cambric of moderately low texture (say 40 picks to the inch) about 6 inches square, and after thoroughly wetting it, put the dough in the middle, tie up tightly with string—allowing plenty of room for the flour to swell—and knead in a basin of water or under the tap until all the starch is washed out.

This point is reached when the water runs perfectly clear from the bag. The kneading must be thorough, but care must be taken that nothing is forced through the bag, which should now contain the gluten.

Open the bag, carefully collect the gluten into one lump, and roll

it between the palms of the hands until it begins to stick. At this point it should be weighed on a small piece of aluminium, and the weight recorded as "wet gluten."

It may now be dried in a steam oven (a process which may take several days), when it will be found that in the wet condition it weighs 2·64 times its dry weight. Consequently, if an early result is required it is usual to weigh wet and divide this weight by 2·64.

The *quality* of the gluten may be determined by stretching the mass as it is being dried between the palms of the hands.

Students are advised to make mixtures of genuine flour with other starches, particularly maize, and note the difference in appearance and adhesiveness of gluten obtained therefrom.

**Softeners.** Ingredients of this class are added to counteract the harshness which would be produced in the fibre by coating it with pure starch. Many fats and oily substances are used, of which the following are important:—

#### TALLOW

Tallow is a well-known natural fat extracted from the sheep or ox. In the animal the fat is contained in little bags called *sacs*, and the tallow-chandler has to get it free from this membrane, which is not composed of fat.

This is termed rendering. The old process was to melt over a fire and press the fat, which will never produce a white tallow—and the method is now practically extinct.

The modern process is to extract with steam at a pressure of about 50 lb. to the square inch. The principle of the method is shown in Fig. 231. The fat is placed in an iron cylindrical chamber, provided with a wooden floor and two doors—one at the top and one near the bottom. Through the chamber runs a pipe that conveys the steam, which escapes from it at intervals. This melts the fat which rises to the top and can be drawn off at the delivery taps. The bottom door is for the removal of the membranous residue. A safety valve is put on the top of the vessel.

By this method, if good and fresh materials have been used, a good and nearly pure tallow, free from dirt and foreign matter, will be obtained. It is evident, therefore, that very little skill and only simple apparatus are required to produce good tallow; but many things affect the quality before it reaches the user, e.g.:—

*Hardness* depends upon the breed, age, food, and sex of the animal. Oil-cake feeding gives a softer fat than grazing the animal.

*Acidity* is the result of age; mutton tallow goes "rancid" sooner than beef tallow of the same quality. Two explanations to account for this acidity have been advanced:—

1. When exposed to air it undergoes change due to the action of ferments, whereby acids are produced.

2. In the presence of light and oxygen (in air) certain constituents of the tallow are oxidized to acids.

At the present time, the second cause is considered to be the more potent.

Water present is largely determined by the honesty or dishonesty of the renderer. The addition of a small amount of caustic potash during rendering—which produces a potash soap—greatly assists the



Fig. 231



FIG. 232

tallow to absorb water as it sets. Such tallow will give an alkaline reaction with the usual indicators.

*Some characteristic Properties of good Tallow.* All pure fats should be odourless, tasteless, colourless, and should not darken when exposed to air. The nearer tallow approaches to these qualities, the purer it is as a rule. All taste, colour, and smell are due to the presence of small quantities of substances other than fat.

Fat should be neutral when in solution, e.g. if some tallow be dissolved in ether, divided into two portions, and a piece of red laemoid paper be placed in one and blue in the other, there should be no alteration in either.

Tallow is completely soluble in carbon disulphide, chloroform, ether, and alcohol. It is insoluble in water, but is capable of absorbing water. If the fat be melted in a long tube which is kept hot by surrounding it with a hot-water jacket (Fig. 232) the two liquids will separate—tallow

\*collecting at the top, water at the bottom. If the tube is graduated it is very easy to calculate the proportion present.

This is due to the fact that, bulk for bulk, tallow is lighter than water. The sp. gr. of tallow is not a constant quantity for all samples.

Beef tallow at 15° C. range from 0.925 to 0.953.

Mutton „ „ „ „ „ 0.937 „ 0.960.

A good average is 0.94.

If determined in the liquid condition at 100° C. and compared with water at 15° C. the range is from 0.885 to 0.863.

The melting-point should be  $111^{\circ}\text{--}113^{\circ}$  Fah. or  $44\text{--}45^{\circ}$  C.

'Beef tallow may range from 42·6° to 50° C. As it gets older it falls, but never below 40° C.

Mutton tallow has a melting-point of about  $47^{\circ}$  C. As it gets older it tends to rise.

*The Testing of Tallow.* A great deal of useful information respecting a tallow can be obtained by performing the following experiments with it:—

1. Find its melting-point. (See Section III, page 28.)
  2. Test its solubility in chloroform or carbon disulphide. Any insoluble matter is an impurity.
  3. Test the solution as obtained above with (a) lacmoid, (b) methyl orange. The latter will detect mineral acids, and the former free fatty acids or alkalies.
  4. Dry and weigh a small evaporating basin half full of small pieces of pumice. Add a few grams of tallow, and heat in a steam oven for several hours until the loss is constant. The loss is due to expulsion of water.
  5. Boil some tallow with dilute nitric acid, cool to solidify the fat, filter and warm some of the filtrate with ammonium molybdate solution. A distinct yellow coloration or precipitate indicates a phosphate—which is due to the presence of bone fat.
  6. Burn a weighed quantity in a crucible and find the percentage of ash present. From pure tallow it is almost nil.
  7. Boil two or three drops of tallow with alcoholic potash solution for several minutes and then add an equal quantity of *warm* water. A white turbidity or precipitate indicates the presence of paraffin oil or paraffin wax, or similar adulteration.

SOAP

Soap is a substance which has been known from very early historical times. We find it mentioned in literature which is quite 2,000 years old, and during recent years a soap factory has been discovered in the remains of Pompeii.

Except that the manufacturer has discovered how to add things which are *not* soap, the process of making is almost identical with that of the ancients.

There are two sorts of soap : (1) Hard or soda soaps ; (2) soft or potash soaps. In all cases it is made by the action of an alkali on a fat or fatty acid or oil. A fat is a compound which can be split up into a fatty acid and glycerine.

The alkali neutralizes the acid in the fat and liberates glycerine. The neutral product is called a soap, which is essentially the sodium (or potassium) salt of the fatty acid.

On a small scale soap may be prepared in the following ways :—

1. Shake up ammonia with olive oil—a white solid results which is largely soap.

2. Make a strong solution of caustic soda ; add this to palm oil, stir well, and allow it to stand. In a short time the temperature rises and solid palm oil soap is formed. This is known as the "cold process" of soap-making.

3. Make a solution of tallow in alcohol, add some caustic soda previously dissolved in water, and simmer on a water bath for half an hour. Add salt to the liquid, and soap is precipitated from solution.

Soaps as now manufactured contain (if unadulterated) from 40 per cent. to 50 per cent. of moisture, from 40 per cent. to 45 per cent. of fatty acid, from 7 per cent. to 10 per cent. of combined alkali, and very little free alkali.

Yellow soaps contain resin, mottled with iron, and an almost endless list of adulterants and additions has been compiled. Starch, clay, talc, chalk, oils, sugar, sulphur, sand, etc., etc., are some of these additions which may be added to produce a soap suitable for some special purpose.

*Pure hard soap* contains 31 per cent. of water—it is impossible to make it with less. If it does not yield this quantity it has been dried since manufacture. Soap-flakes often contain less than 10 per cent.

*In cocoa-nut oil soap* the water may reach 75 per cent. to 80 per cent., and it may still appear a fairly solid soap.

The analysis of a good sample of soap yielded the following results : 55 per cent. fatty acid, 9 per cent. fixed alkali, 36 per cent. glycerine and water.

The value of a soap is largely determined by the quality and quantity of fatty acid present ; any hard soap with more than 64 per cent. has been dried, any with less has been intentionally reduced.

Free alkali, which may be detected by adding a drop of phenolphthalein to a freshly cut surface, when a pink colour is produced, is not—except that it indicates a badly made soap—very objectionable

from a sizing point of view. In fact, a slight alkalinity in soap will neutralize undesirable acidity in a rancid tallow.

The chief reason for using soap in a size mixing is that its presence assists in the more perfect emulsification of the fat and thus a more uniform liquid is produced.

Some sizers use no soap *as such*, but they add a small amount of caustic soda. During boiling this alkali reacts with some fat to form soap, and therefore they are using it although not adding it as a separate ingredient. Soap is also present in many trade softeners and sizing compositions.

Soap must not be used in the presence of metallic chlorides or some of the value of each is destroyed.

For sizing purposes a good soft soap or a hard soap made by the cold process is to be preferred to an ordinary hard soap, as soft soaps and cold-process ones often contain all the glycerine present in the original fat.

*The testing of soap* should include these determinations :—

1. Water. Take a sample from the middle of the bar if hard, or below the surface if soft. Weigh quickly on a tared watch glass and then shred it if hard soap, and dry in an air oven at a temperature of  $105^{\circ}\text{C}$ . till no further loss in weight occurs. Calculate to a percentage.

2. Find the amount of ash. (See Section III, pages 32 and 33.)

3. Fatty acid. Weigh out 25 grams of the sample and dissolve in a beaker of water *on a water bath*. When it is near boiling-point add a few drops of methyl orange, and then strong hydrochloric acid till the indicator has been turned a distinct pink colour. This liberates the fatty acid. Boil gently till the acid forms as an oily layer on the top of the liquid.

Add 5 grams of stearic acid or paraffin wax, warm up until the two are thoroughly mixed, and then allow the vessel and contents to cool. Carefully remove the cake, dry on filter paper, and weigh. Deduct the weight of wax (which was added to ensure that it set solid) and calculate to a percentage on the amount of soap used.

### GLYCERINE

This substance has a certain application in sizing—less, probably, than it deserves—as besides acting as a softener it is hygroscopic and has mild antiseptic properties.

Glycerine may be obtained from fats by subjecting them to the action of superheated steam at a temperature of  $300^{\circ}\text{C}$ ., but as a rule most commercial glycerine is obtained as a by-product in the two industries of soap-making and candle-making.

This glycerine is often contaminated with many undesirable substances, and is very dark in colour—sometimes almost black—

and although then cheap, it is useless to the sizer on account of the darkening effect it would produce in the size.

If, however, it is only slightly brown it may be used, provided certain impurities are absent.

As good glycerine is expensive, many "glycerine substitutes" are on the market; these are often only solutions of glucose sugar, and are almost useless for sizing purposes.

Glycerine mixes in all proportions with water and alcohol, but is insoluble in carbon disulphide and chloroform. Taste is a very good test to apply to glycerine—if impure, it is distinctly unpleasant.

A good sample of commercial glycerine yielded the following results on analysis:—Sp. gr. 1·3, 80 per cent. to 82 per cent. real glycerine, 10 per cent. ash, and gave no precipitate on being added to strong hydrochloric acid.

*The Testing of Glycerine.* Sufficient information as to its suitability for use as a sizing ingredient will be obtained by performing the following experiments:—

1. Find its sp. gr. (See Section III, pages 29 and 30.)
2. Add some to an equal volume of strong hydrochloric acid in a test tube. Invert the tube two or three times to thoroughly mix the two liquids, and then allow the mixture to stand for half an hour. If at the end of that time no white precipitate has been deposited, it may be assumed that salt is not present in sufficient quantity to condemn it.
3. Test for presence of glucose by diluting with an equal volume of water and then boiling it with some Fehling solution. If sugar is present the blue colour is destroyed, and a red precipitate is produced.
4. Test for lime by adding some crystals of ammonium oxalate to some which has been diluted with twice its own volume of water. Shake well at intervals—a white precipitate indicates the presence of salts of lime.

## WAXES

Chemically, waxes are quite distinct from fats, but the classification is not based on their physical state, e.g. Japan wax is really a fat, and sperm oil is a wax.

Of the substances commonly known as waxes, the ones used in sizing are:—Japan wax, paraffin wax, spermaceti, and wool grease.

Japan wax and spermaceti are both expensive substances and are used in very small quantities in mixings; their use seems to be "faddy" rather than essential in many cases, but spermaceti wax and paraffin wax crystallize from tallow and in some pure mixings are used by reason of this property, thereby a peculiar feel and appearance are obtained.

Japan wax has a high melting-point and is sometimes used for cloth sent to hot and very humid countries, e.g. Java.

Paraffin wax and wool grease are much more widely used, and in some cases they are very desirable or even necessary ingredients, but the former is a very dangerous ingredient to put into size if the cloth is to be afterwards bleached or dyed.

Because of the extraordinarily high price of tallow now prevailing, many manufacturers have been induced to use other forms of grease. One of the most successful has been wool grease, the best qualities of which are not usually sold under that name.

This substance is excreted through the skin by sheep and collects in the wool by absorption. In wool washing it is extracted in a very impure condition. When highly purified a very valuable neutral wax is obtainable which is sold under the name of *Lanoline*, and has been largely used in the preparation of ointments, due to the very characteristic property it possesses of being readily absorbed by the skin.

The crude "recovered" or "Yorkshire" grease is a mixture of free and combined fatty acids and alcohols. It is a dark yellow or brown viscous substance of melting-point 39° C. to 42° C., sp. gr. 0.973 at 15° C., and has a distinct smell of sheep.

#### CHINA CLAY

Of all weight-giving substances used in sizing none is so successful as good China clay.

Magnesium sulphate, gypsum, barytes, and other compounds have been used, but it has been demonstrated to the sizer that it rarely pays to use them except for the more common kinds of cloth.

In nearly all parts of this country clay is found in the soil, but in only a few districts is it of the kind necessary for the sizer's use, i.e. kaolin or China clay.

This kind is found in geological deposits in Cornwall and Devon, where older rocks have been weathered and destroyed, and the small particles of aluminium silicate have been collected by the action of water. The deposits have become dry, and thus form the natural beds of clay.

These beds contain particles of sand, mica, and iron salts which were present in the original rock. The method of treatment is to mix up the mineral with water: the clay, being lighter than sand, remains in the top layer of liquid, which is run off and then allowed to settle. If this is repeated several times a clay free or nearly free from impurities is obtained. It is then dried in kilns.

Physically, clay is a very fine white powder which has a great capacity for absorbing water, and, owing to this absorption and its

fineness, becomes plastic. It is soft and soapy to the touch, and when breathed upon it emits a characteristic earthy odour.

For sizing purposes it should be free from iron, grit, and lime. It should possess also an unctuous feel—plasticity is not the quality desired. It should not be coloured artificially.

The amount of moisture present in commercial China clays often varies very considerably. This is due generally to imperfect drying; but even if the clay be thoroughly dried at steam heat, a certain amount of water remains, varying from 10 per cent. to 12 per cent., which is only expelled at red heat.

For heavy sizing especially, it is very desirable that the sizer should know the excess of moisture above this 12 per cent., which might be called the "strength" of the clay. This may be determined by drying in a steam oven, or better, at 105° C. to 110° C. for several hours, until the loss is constant. An aluminium tray is a suitable receptacle to use for the purpose.

The sizer whose speciality is heavy sizing would do well to determine the percentage of "free" (i.e. expelled at 105° C. to 110° C.) and "combined" (expelled at red heat) moisture in his various samples of clay. An example of this is given on page 33.

Mellor states that the best temperature at which to determine "hygroscopic" (i.e. free) moisture is 109° C. to 110° C. For the loss on ignition (i.e. combined moisture) for Cornish China clays, previously dried at 110° C., he gives the following figures:—

Maximum . . . .	14.12 per cent.	These calculations are made on the <i>dried</i> , not natural, clay.
Minimum . . . .	11.03 , , ,	
Mean of six . . . .	12.5 , , ,	
Ideal . . . .	14.0 , , ,	

#### LABORATORY EXERCISES WITH CHINA CLAY

1. Test for chalk by adding hydrochloric acid. If present, effervescence will result. Filter, and to the filtrate add ammonium oxalate—a white precipitate is obtained if chalk, or plaster of paris, or gypsum is present.

2. Grit should be detected by shaking a few grains with water, allowing the mixture to stand for two or three minutes, pouring off the top layer, and examining the sediment. This may be done by rubbing it between two glass microscope slides or by examination under the microscope.

3. The presence of artificial colouring may often be detected by adding a few drops of strong ammonia and stirring with a glass rod.

4. Boil some with hydrochloric acid, filter and divide it into two parts. To one add potassium ferrocyanide—a blue colour is produced

If iron is present, the depth of tint depending upon the quantity in solution. A clay suitable for sizing purposes will show only a faint colour.

To the other portion add ammonia and boil. A reddish brown precipitate indicates the presence of an undesirable amount of iron in solution.

### METALLIC CHLORIDES

These substances are used to give either weight or antiseptic properties, or both, to the twist.

Those most frequently used are (1) zinc chloride ; (2) magnesium chloride ; (3) calcium chloride.

Beside these, another sometimes gets into the size—due to adulteration of ingredients—i.e. (4) sodium chloride.

Of these, the *only antiseptic* is zinc chloride. Zinc chloride, magnesium chloride, and calcium chloride are all deliquescent bodies (i.e. they abstract moisture from damp air).

Calcium chloride and sodium chloride are not desirable ingredients to have in size except in very small proportions.

**Zinc chloride** is made on the commercial scale from scrap zinc or zinc ashes and skimmings or compounds of the metal that have been produced as by-products in certain manufacturing processes, by mixing the raw material with hydrochloric acid. The resulting liquor is treated to free it from iron and other undesirable impurities and concentrated to a syrup-like mass containing about 45 per cent. of anhydrous chloride of zinc, having a sp. gr. of 1.51-1.52 (102-104° Tw).

For export it is usually evaporated till nearly all the water is expelled and it sets as a white solid containing zinc equivalent to 98 per cent. or more of zinc chloride.

Commercial zinc chloride is seldom pure, as the cost of removal of *all* impurities would make it a very expensive chemical ; nor is it necessary for sizing purposes that this highest degree of purity be attained. It is sufficient as a rule that free mineral acid and iron be absent, and that less than 1 per cent. of sodium chloride be present.

The tests for these impurities may be conducted in the following manner :—

1. **Salt or Sodium Chloride.** Pour some into a test tube half full of strong hydrochloric acid, and after mixing allow it to stand for half an hour. If salt be present to a greater extent than 1 per cent. it will be precipitated in small white crystals.

2. **Iron Salts.** Boil a few drops with *pure* nitric acid and then add one drop of it (in the end of a glass rod) to some potassium thiocyanate solution in another test tube. The production of a blood-red colour shows the presence of iron. The depth of the colour depends

upon the amount of iron in solution—if it be but faint the sample may be passed as fit for use.

**3. Free Acid.** The indicator used must be either Congo red paper (which is turned blue), or methyl orange solution (which is turned pink) by free mineral acid. As a rule the manufacturer tries to produce a solution which is slightly basic in character, that is, it contains a little oxychloride of zinc in solution.

#### LABORATORY EXERCISES

**I.** Examine samples of zinc chloride, (*a*) solid, (*b*) in solution; and perform the following experiments with them:—

##### *Solid*

- 1. Expose to air on a watch glass for half an hour; note what happens.
2. Test solubility in a small quantity of water.
3. Note effect of adding more water to this; then addition of dilute hydrochloric acid.
4. Take a small piece in a porcelain crucible; heat strongly and note all changes.

##### *Solution*

1. Test for zinc by adding ammonia and ammonium sulphide.
2. Test for salt by adding some to an equal quantity of strong hydrochloric acid.
3. Test reaction to litmus, methyl orange, and Congo red.
4. Test for presence of iron by potassium thiocyanate.
5. Test for calcium by adding ammonium chloride, ammonia, and ammonium oxalate.

**II.** Heat some zinc oxide on charcoal with the mouth blow-pipe, using the oxidizing flame. Note colour—hot and cold. When cold, moisten residue with a few drops of cobalt nitrate solution. Reheat, and again note the colour. Repeat the experiment, using in turn, on a fresh spot on the charcoal: alumina, magnesia, clay, “antiseptic,” “zinc,” “septic.”

**III.** Prepare a solution of zinc chloride by dissolving zinc powder or granulated zinc in commercial hydrochloric acid, using excess of the metal. When action has ceased filter through glasswool (It will dissolve filter paper) and concentrate to a syrup. Find its specific gravity, and test it for the presence of free acid and iron.

**Magnesium Chloride.** This substance is a white crystalline, very deliquescent salt, the chief source of which is the enormous Stassfurt deposit in Germany—not far from Jena. From these mines it comes into commerce in an exceedingly pure condition.

In our own country there are large deposits of dolomite and magnesium limestone—which are compounds of lime and magnesium carbonates. Upon treating with hydrochloric acid the carbonates are converted into chlorides. The magnesium chloride is less soluble than

the calcium (lime) chloride, and is crystallized out first. By this method of preparation the chloride of magnesium always contains chloride of calcium as an impurity.

It can be detected by adding to a solution of magnesium chloride the following solutions in the given order:—Ammonium chloride, ammonia, ammonium oxalate. Gently warm. A white precipitate is formed if calcium chloride is present.

Magnesium chloride is cheap, very deliquescent, and thus gives weight to the yarn or cloth, but it must not be used in the presence of soap, or without the addition of an antiseptic, as magnesium chloride itself is *not* one.

Calcium chloride must not be confused with bleaching powder—which is not, strictly, chloride of lime at all, although often so called. This is not a suitable compound to use in size mixings—lime salts never are. It is very cheap, being formed as a by-product in many chemical industries, and thus it is often used to adulterate other chlorides.

It is very deliquescent, but has no antiseptic value, and it must not be present in mixings that contain soap.

#### FERMENTATION, MILDEW, ANTISEPTICS

For perhaps thousands of years it has been known that if a sugar solution be exposed to air and warmth it is gradually converted into a liquid having very different properties, and that if this liquid be further exposed it becomes sour.

Later it was noticed that bubbles were formed during the process, and hence arose the term fermentation. Attempts to explain why wine was converted into vinegar were made as early as 1670; and Dr. Willis (who died in 1675) considered that all vital actions were due to different kinds of fermentation.

Liebig investigated many cases of fermentation and came to the conclusion that the process was due to the action of ferments. It remained for Pasteur to experiment exhaustively in the subject, and as a result of his researches he advanced the view that fermentation was the result of vital action.

Later investigators have shown that both causes operate. When starch is taken into the mouth and mixed with the saliva excreted from the glands of that organ, it is thereby brought into contact with a ferment known as ptyalin, which at the temperature of the body, converts the starch into sugar.

A similar ferment is present in the growing barley grain, and if barley is kept warm and moist, sugar is formed. It is the same with other grains—the nature of the changes and substances produced being determined by the character of the particular ferment.

When milk goes sour it is due to the fact that fermentation has taken place and has produced some acid. Pasteur showed that if milk is kept out of contact with air it could be preserved for months without turning sour. Tyndall proved that it was only necessary to "filter" the air that surrounded the milk and it could still be kept sweet. It was demonstrated that the necessary agent to set up milk fermentation was a "germ" from the air.

The same cause explains the fermentation of sugar, but here we have a simple cell (yeast). And in the case of vinegar we have a similar organism—the *Mycoderma aceti*.

Spores (or seeds) from certain other plants—still low in the scale of life, but higher than those already mentioned—are always present in air and are ever seeking a "soil" suitable for their development and growth. The materials that are required by fungi for luxurious growth are ammonia and phosphates.

The common name for these is *mildew*—the botanical, fungi; and the microbiologist has identified thousands of different species.

Then there are other bodies known as *bacteria*, which feed and multiply in numberless media; and many virulent diseases are attributed to their action, e.g. cholera, plague, lockjaw (tetanus), yellow fever, typhoid fever, etc. In fact this world teems with life of all sorts, the "low" type being much more plentiful and prolific than the "higher" forms.

Given certain conditions, the chief of which are warmth, moisture, and suitable food, they will flourish and multiply at an enormous rate. But if they do not multiply, the most virulent of them appear to be harmless and even unappreciated by the ordinary senses.

Now to apply these facts to explain fermentation and mildew as met with in the cotton industry.

When flour is mixed with water and exposed to the air it rapidly comes into contact with certain germs, or it may contain ferments derived from the natural grain. These commence to convert the gluten which is present in the flour into small quantities of other chemicals, probably carbon acids, ethers, alcohols, etc. Pure starch will not ferment because there is no plant food in it suitable for the growth of germs.

Sizers know that flour which has been fermented for a reasonable time is less liable to mildew than a paste which has not been fermented. The explanation is that some of the substances which are the products of fermentation are slightly antiseptic in their nature.

An antiseptic is a substance which by its presence prevents the growth of low forms of animal and vegetable life. Many substances are known to act in this manner, amongst which are zinc chloride, mercuric chloride, copper sulphate, carbolic acid (phenol), iodiform, formalin, glycerine, etc.

**Mercuric chloride**, or corrosive sublimate, is the most efficient, but so deadly poisonous in its effects on the human system that it must not be used for trade purposes on any account. It receives its application for sterilizing in typhoid fever and cholera, the virulent germs of which it is able to destroy thoroughly.

**Iodoform** is much too expensive for general use; it is used in surgery.

**Glycerine** is fairly efficient if the cloth is not subjected to very damp conditions, but it is not suitable for heavy sizing.

**Formalin** or **formaldehyde** is effective under certain conditions as a preventive of mildew, but it is much more effective against putrefactive bacteria. It must be remembered that this chemical is very volatile, and is lost by boiling. It is very effective for fumigating a room in which a scarlet-fever patient has been living, but to claim that it is equally active in the destruction of mildew spores (as was suggested in the celebrated "weavers' cough" epidemic at Burnley a few years ago) is claiming too much for it.

**Carbolic acid** is prepared from coal tar and is purified by recrystallization. It is possible thus to obtain a very pure product. This quality is rather expensive for sizing purposes, but stains may result on the cloth with less pure grades. Carbolic is a most efficient antiseptic for prevention of bacterial growth, but to prevent mildew it must be actually in contact with the material in the solid or liquid condition; its vapour is not equally effective with respect to the prevention of fungoid growth. Another objection to phenol is its characteristic odour, which is not liked in cloth.

**Copper sulphate**, or **blue vitriol**, is a cheap and well-known chemical which has great power as a fungicide, in fact perhaps the best that is available at the present time; but for textile purposes the quality used in agricultural spraying mixtures is unsuitable. It is essential to use a grade that contains but a trace of iron salts, and it must also be free from uncombined sulphuric acid.

There are other objections to its use, such as liability to produce copper stains, and its action as a catalyst (page 70) in the presence of certain other textile materials.

**Zinc chloride**, first suggested by Sir W. Burnett, and consequently sometimes known as **Burnett's disinfecting fluid**, is, *generally speaking*, the most satisfactory antiseptic for cotton goods. The substance is sometimes called "antiseptic," which is not desirable, for, as we have already noted, this is a name used for the whole class of substances.

Zinc chloride is deliquescent as well, and probably this property has had something to do with its popularity, but for heavy sizing it stands unrivalled at the present time. For certain goods and in special circumstances it is inadmissible.

In these circumstances it is often difficult to recommend a suitable substitute, but salicylic acid is sometimes permissible. This is another very efficient fungicide, but it will change the colour of certain direct dyes such as Congo red, and in that event it would be better to use sodium salicylate, which however is, weight for weight, only about half as efficient.

From this short account of antiseptics it will be seen that the question "Which is it advisable to use?" is often by no means an easy one to answer.

The action of antiseptics has not been *thoroughly* explained; all that can be definitely stated is that they appear to be substances which are capable of either killing low forms of life or bringing about suspended animation.

In their presence, even if all the conditions conducive to a successful growth are fulfilled, multiplication does not take place or is very considerably retarded.

Some chemicals are more potent in this direction than others, and thus we find that ultimately fermented flour will mildew in spite of the presence of the antiseptic bodies which have been produced.

In the case of dry starch, it is found that no fermentation takes place, but a *starch paste* will mildew. All natural starches contain some nitrogen compounds, and the plant probably first feeds on these later the constituents of the starch, in conjunction with the nitrogen of the air, form a sufficient soil.

The action of caustic soda as a "preservative" (it can hardly be called an antiseptic) when boiled with the starch to form the paste is probably due to its destructive action upon nitrogenous matter, so reducing the available food.

Tyndall showed conclusively that moisture was absolutely necessary to fungoid growth. A substance which is perfectly dry will never mildew, but add water to it even in small quantities (i.e. in the form of moisture), and it is *liable* to mildew at any time.

Now size and sized goods always contain water. Cotton itself is hygroscopic, and will abstract moisture from the air, and deliquescent bodies like calcium and magnesium chlorides add greatly to the liability. Therefore the greater the amount of water or deliquescent present in the size or on the yarn and cloth, the larger is the amount of antiseptic required in order to prevent the formation of mildew.

#### LABORATORY EXERCISES IN THE TESTING OF "RESIDUES"

You are provided with samples of typical ashes of various sizing ingredients, etc., labeled A to H. Examine them as instructed below:

A (hard soap). Add a few drops of dilute hydrochloric acid. Test gas evolved for carbon dioxide. Dip a platinum wire in the liquid

and test for sodium in the flame (yellow). What was the probable composition of the ash, and was it completely soluble in acid?

B (soft soap). Repeat as for A, and in addition dissolve some in dilute nitric acid. Concentrate and crystallize. Note shape of crystals. Are they sodium nitrate or potassium nitrate, or both?

C (glycerine). Extract the portion soluble in water, and test it for salt by adding a few drops to strong hydrochloric acid. Add dilute hydrochloric acid to some more and test in the flame for calcium (red). Also test the solution with ammonia and ammonium oxalate. What was the ash?

D (size). Extract portion soluble in water and test it for sodium carbonate as in A above. Extract the residue with dilute hydrochloric acid and test the extract for magnesium with ammonium chloride, ammonia and sodium phosphate (white precipitate). Test the residue from second extraction for clay by heating on charcoal with blowpipe, moistening with cobalt nitrate, and reheating (blue mass).

E (sago). Test solubility in dilute acids, including aqua regia. Fuse some with fusion mixture, lixiviate, and test solution for silica with hydrochloric acid (gelatinous precipitate). Also test grittiness between two pieces of glass.

F (cloth). Dissolve in dilute hydrochloric acid and add ammonium chloride and ammonia (precipitate = aluminium). Filter, and to filtrate add ammonium sulphide (precipitate = zinc). Filter, and to filtrate add ammonium carbonate (no precipitate = absence of calcium). Then add sodium phosphate (precipitate = magnesium).

G (clay). Test this for:—

(a) Carbonate, with dilute hydrochloric acid.

(b) Calcium, by flame reaction.

(c) Bleaching powder, by mixing some with starch paste. Then add a little acetic acid, followed by two or three drops of potassium iodide. (Blue colour of iodine is liberated by chlorine evolved from bleaching powder.)

H (dressing material). Extract with water and test separate portions for:—

(a) Copper. Addition of ammonia produces blue colour.

(b) Sulphate. Barium nitrate gives a white precipitate insoluble in nitric acid.

(c) Magnesium. White precipitate with ammonium chloride, ammonia, and sodium phosphate.

Test the well-washed residue from the water extraction for barium in the flame, after moistening with hydrochloric acid.

time, keeping the volume constant. Filter: a dark red liquid is produced. Dilute it till correct sp. gr. is obtained.

**4. Prussian Blue.**

Dye the cotton iron buff and then pass it through a solution of potassium ferrocyanide acidified with sulphuric acid.

**5. Manganese Bronze.**

Impregnate the yarn with a solution of manganeseous chloride. Fix it in a *hot* solution of caustic soda containing 30 grams per litre.

Rinse it in a weak solution of bleaching powder (strength 1° Tw.).

Wash and dry.

### TESTING DYED SAMPLES

It is very essential that every dyed sample should be submitted to certain tests, and that a systematic record of the same should be preserved. The plan illustrated on the next page will be found a suitable one for beginners.

To carry out the tests proceed as follows:—

**1. Fastness to Light.** Take a piece of glass about 4 inches by 6, and cut a piece of white cardboard the same size. Bind them together along one edge by means of a strip of photographic adhesive, "leatherette." On the top of the cardboard put a piece of black paper, on the top of this a piece of white filter paper, and on the top of this a few strands of the dyed yarn which is to be tested.

Cover half of it with two thicknesses of black paper and let the glass fall into position.

The remaining three edges can now be bound, or two rubber bands can be passed round.

The whole arrangement can now be exposed to *bright direct sun-light* for days, or weeks (if necessary).

The degree of fastness is judged by comparing the portion exposed with the portion that was kept under the black paper.

Generally speaking, sulphur and mineral dyes are fast to light; basics very fugitive (particularly the lighter shades), and directs vary considerably.

**2. Fastness to dilute Acids and Perspiration.** Steep in cold 25 per cent. solution of acetic acid for five minutes. Wring, wash, and dry.

**3. Fastness to Washing.** This test can be made in two ways:—

(a) Steep for five minutes in a 1 per cent. solution of sodium carbonate.

(b) Plait with a few threads of undyed yarn and boil for ten minutes in a 1 per cent. soap solution. If the colour "bleeds," the undyed yarn will be tinted, and it should also be filed in the record.

## SPECIMEN PAGE OF RECORD

Name of Colour used .....				
Class of Dye: Substantive or Direct Cotton Colour.....				
Shade	Light	Medium	Dark	Exhaust
Percentage Dye used .. .	—	—	—	—
Percentage Glauber or Salt ..	—	—	—	—
Percentage Soda Ash .. .	—	—	—	—
	O	O	O	O

Samples of above, showing fastness to :—

1. Light . . . . .	O	O	O	O
2. Dil. Acids . . . . .	O	O	O	O
3. Washing . . . . .	O	O	O	O
4. Stripping . . . . .	O	O	O	O
5. Bleaching . . . . .	O	O	O	O

Remarks .....

4. *Fastness to "Stripping."* Plait with white yarn and boil in pure water for fifteen minutes. Look for (a) tinting of white yarn ; (b) coloration of water ; (c) loss of colour on the dyed yarn.

5. *Fastness to Bleaching.* There is considerable misconception with respect to what is known as "fastness to bleaching" and "bleaching colours." Almost any colour can be wholly or partly bleached if the bleaching process be intense enough. Modern laundries as a rule use bleaching chemicals in a manner which acts much more drastically than is the case in ordinary calico-bleaching, and it is unreasonable to ask for fastness to bleaching in an unlimited sense.

The test here given is a reasonable one, and is similar to that applied by one of the largest manufacturers of "cloths to stand bleaching" in Lancashire.

Make a solution of *fresh* bleaching powder, strength 5 grams per 100 c.c., and filter.

Steep the dyed yarn in the (cold) filtrate for ten minutes.

Remove, and without squeezing or washing put it in dilute acetic acid or dilute sulphuric acid (1° Tw.) for ten minutes.

Remove, wash well under running water, and dry in the air.

## SECTION XVIII

### MERCERIZING

#### MERCERIZATION OR MERCERIZING OF COTTON

This word was coined from the name of the discoverer of the phenomenon.

John Mercer, whilst experimenting in 1860 with caustic soda solution and cotton yarn, found that if the concentration reached about 20 per cent., and the fibre was steeped in it for 5 to 10 minutes, and afterwards removed and thoroughly washed, certain very noticeable changes had been produced :—

1. There was a shrinkage in length, varying between  $\frac{1}{2}$  and  $\frac{1}{4}$  of the original.
2. If the cotton was dried the weight was greater than that of the original by approximately 5 per cent.
3. The strength of the yarn was also increased by anything up to 60 per cent.
4. The fibre was made "fuller."
5. It showed an increased affinity for dyes.

This effect was not confined to cotton in the form of yarn—similar results could be produced in cloth.

Mercer patented his process with the idea of putting on the market a stronger and fuller yarn and cloth.

Unfortunately mercerized cotton was not a commercial success in Mercer's lifetime, and in fact made very little progress until another property regarding it was discovered some thirty years later. Since then it has increased enormously in popularity.

This important characteristic is produced by stretching the cotton during or after immersion in the alkali, and keeping it so during the washing process.

The fibre is thus prevented from contracting, with the result that an external lustre is produced.

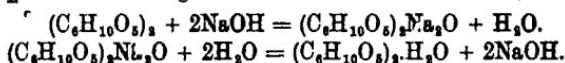
It is true that the increase in strength is reduced—being less than 40 per cent. instead of 60 per cent., but the "silky" effect obtained more than counterbalances this deficiency.

The chemistry of the process as worked out by Gladstone is :—

- While the cotton is immersed in the soda solution a compound of cellulose and sodium oxide is formed.

During the washing this is decomposed by the removal of the sodium and the substitution of hydrogen (from the water) in its place. This results in the production of a hydrate of cellulose.

Assuming the empirical formula of cellulose to be  $(C_6H_{10}O_5)_n$ , we can represent the changes as follows :—



Mercerized cotton..

The examination of mercerized cotton under the microscope shows that the fibre has been somewhat untwisted, the walls being considerably increased in thickness, the hollow flattened ribbon being changed to a thickened cylinder with practically no hollows (Fig. 240).

Poor-quality or short-staple cotton is not suitable for mercerizing, and the best results are obtained by using 2-fold Egyptian or Sea Island which has been previously bleached.



Fig. 240

The strength of caustic soda used should be between 50°-70° Tw. and the operation should be conducted at a temperature of 60° Fah. After washing with water the lustre may be increased by washing with dilute acetic acid and the silky effect may be still further increased by a special calendering process.

The best chemical test to apply to yarn or cloth to detect mercerization is to treat the cotton with a cold saturated solution of zinc chloride, potassium iodide, and iodine.

The reagent is prepared by dissolving  
30 grams zinc chloride (pure solid)  
5 " potassium iodide                          } in 24 c.c. of water.  
1 gram iodine

It should be kept in a small glass stoppered bottle.

If the sample is white it may be used without previous preparation. If it is coloured it must be first bleached and dried before the test is applied.

A very small piece (if cloth) or a few strands (if yarn) are immersed in the dry condition for 2 or 3 minutes in the liquid, and then transferred by means of a glass rod to an evaporating dish nearly full of water. By means of the rod the cotton is kept under the surface of the water and moved about to wash it.

If the cotton has not been mercerized the dark blue colour will gradually become fainter and ultimately disappear. In the case of mercerized cotton the colour remains a distinct blue.

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